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Corrosion

Official Publication
NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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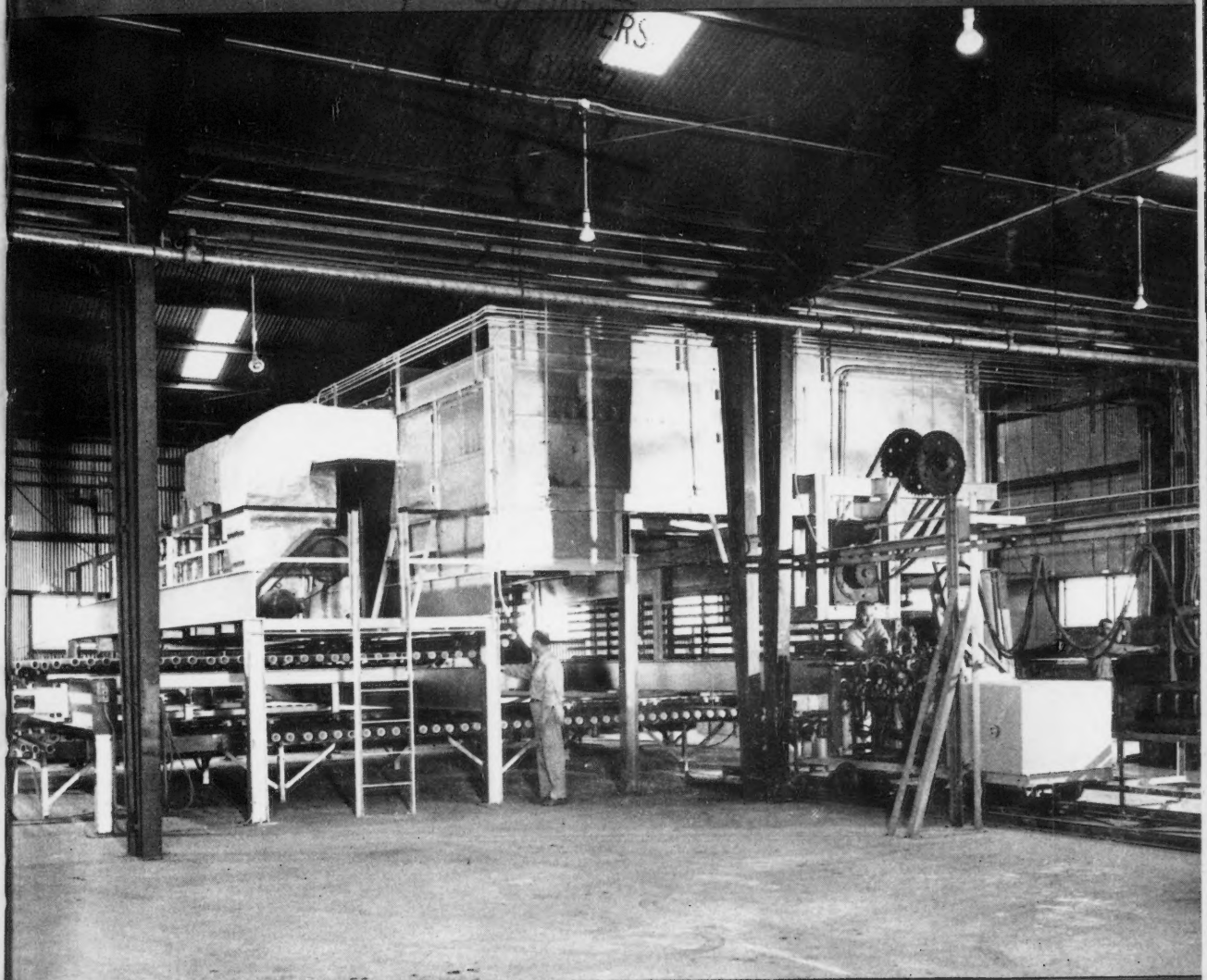

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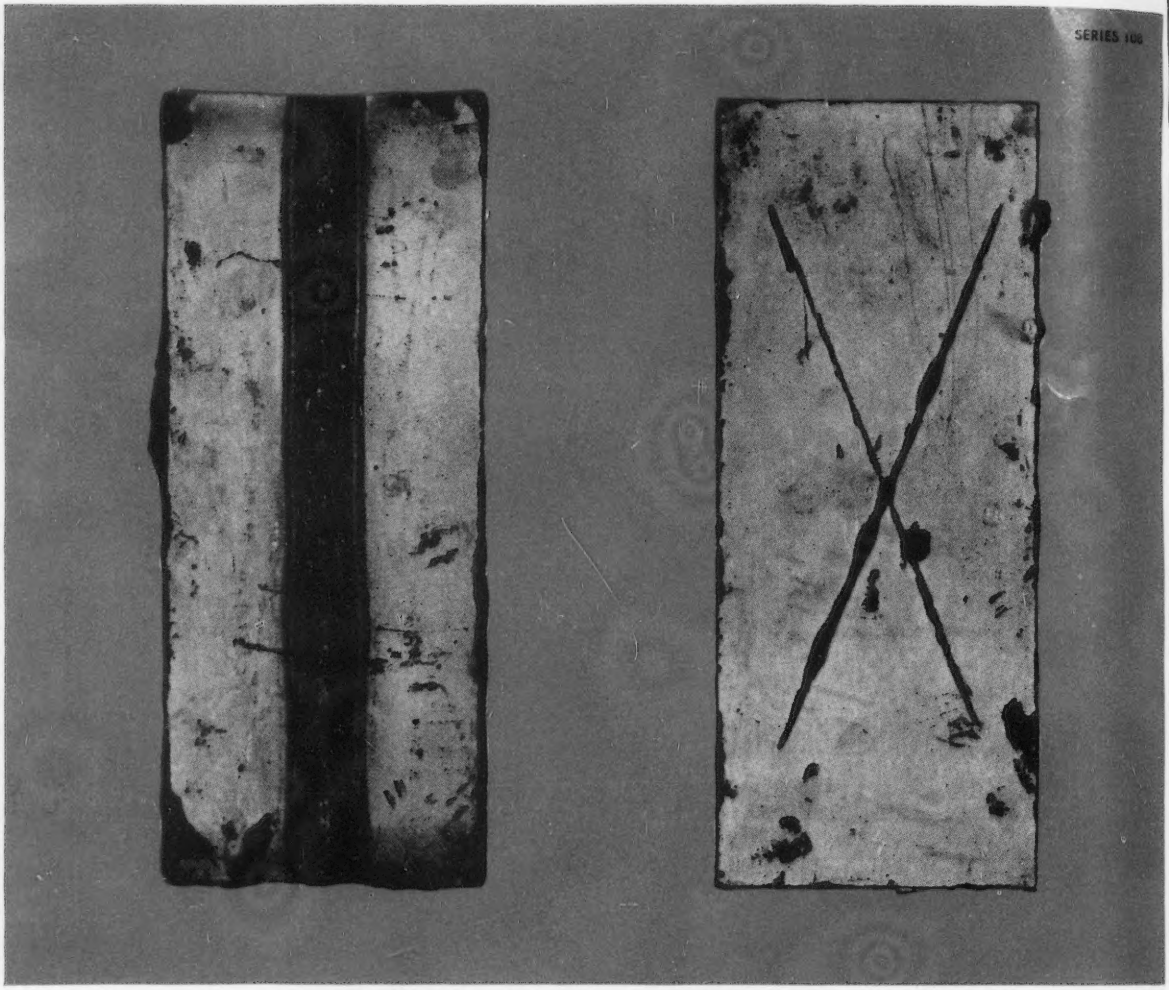

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No. 87 combines the time-tested chemical and weather resistance of vinyl coatings with the thickness of mastic, yet it is easily sprayed with standard equipment over a suitable primer. Available in white, gray, black and aluminum. Use it for enduring protection at lower cost per square foot per year. Complete details mailed on request.—Amercoat Corp., South Gate, Calif.

AMERCOAT Corporation, Dept. GI
4809 Firestone Blvd., South Gate, Calif.

Please send me your latest information on Amercoat No. 87.

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Position_____

Company_____

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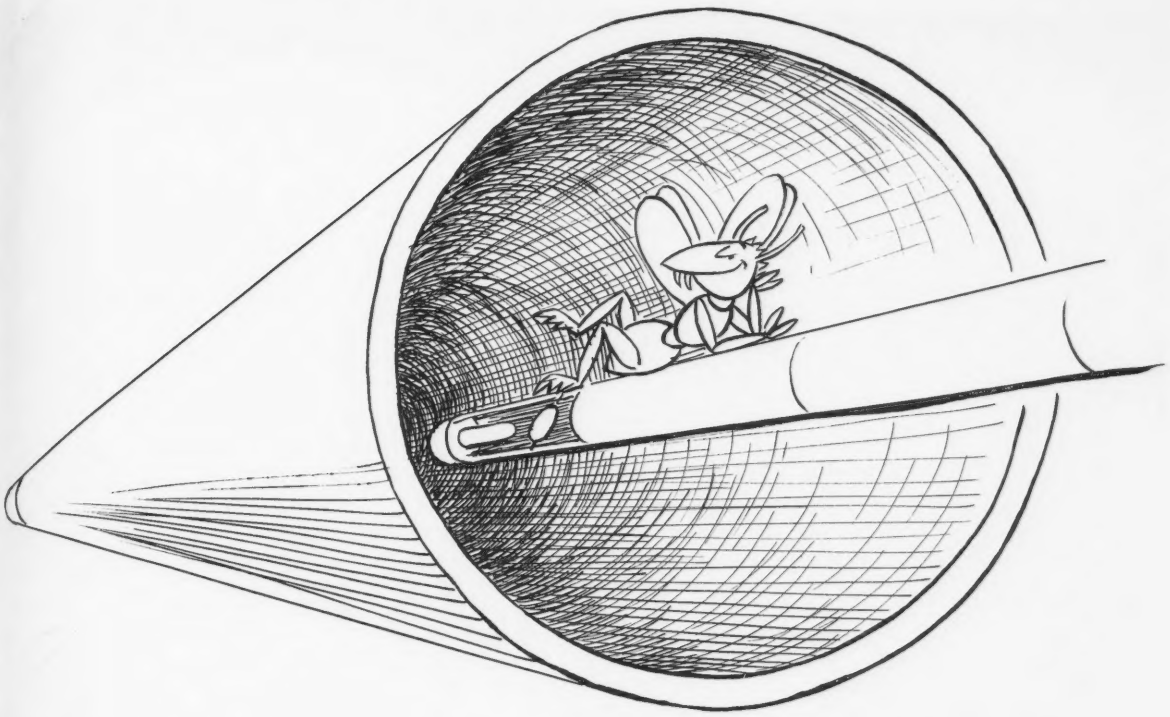

IS YOUR INSPECTION SERVICE MISSING SOMETHING?

Seriously, many a dangerous "bug" escapes ordinary inspection techniques. Plastic Applicators' inspection procedure—after descaling, degreasing and sand blasting to bare metal—repeatedly has turned up hidden defects exceeding API specifications in previously field-inspected and approved tubular goods.

Plasticap offers you a choice of optical and magnetic inspection and hydrostatic testing. All joints are tested with API drift for diameter and straightness; upset areas on grades above N-80 are blasted to white metal. Materials to be coated are handled only once and may be stored in our yard until required.

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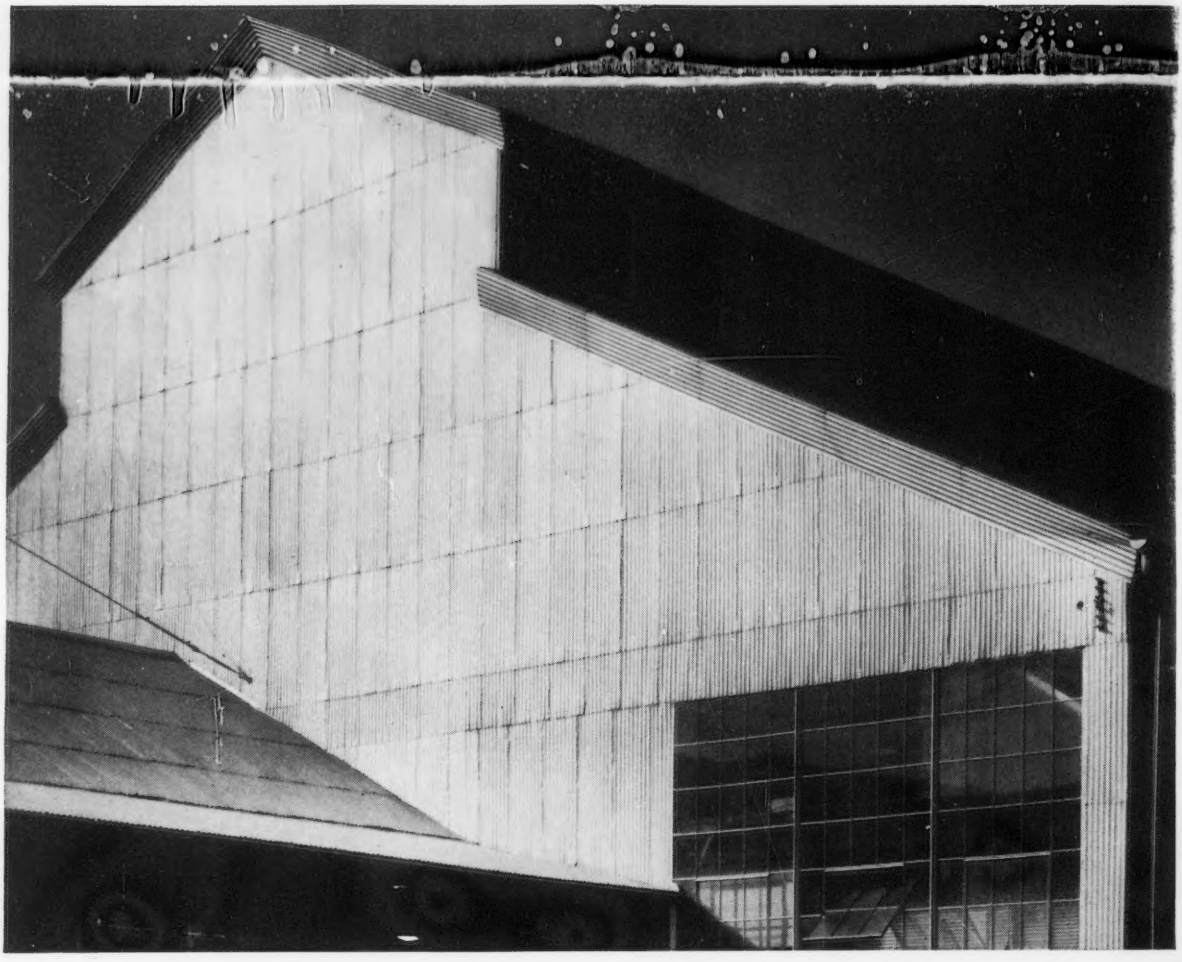

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that develops an excellent zinc phosphate coating on mill passivated galvanized stock

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If you are having trouble getting a good zinc phosphate coating on your galvanized stock, consult us. We'll be glad to send you descriptive literature or have a technical representative call.

AMERICAN CHEMICAL PAINT COMPANY, Ambler 40, Pa.

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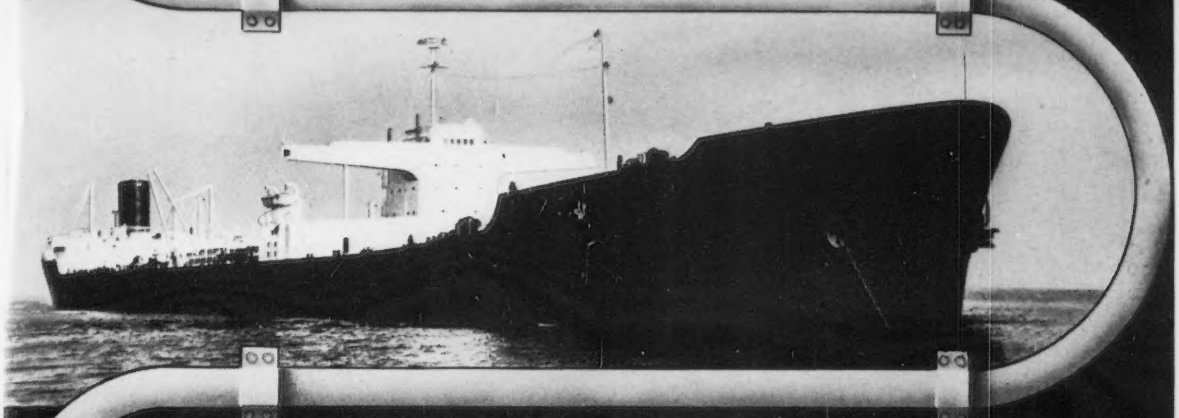

from corrosive contact of hot, sour crude oil and salt water ballast, the Niphos-coated heating coils of National Bulk Carrier's newest tanker promises to outlast ordinary coils by several years!

The new Niphos Process, exclusive with Tube Reducing, makes *any* ferrous metal as corrosion-resistant as high-priced stainless for many applications. Applied on any shape — fused and alloyed in place by heat treating — Niphos-clad metal can outlast untreated ferrous as much as 10 to 1!

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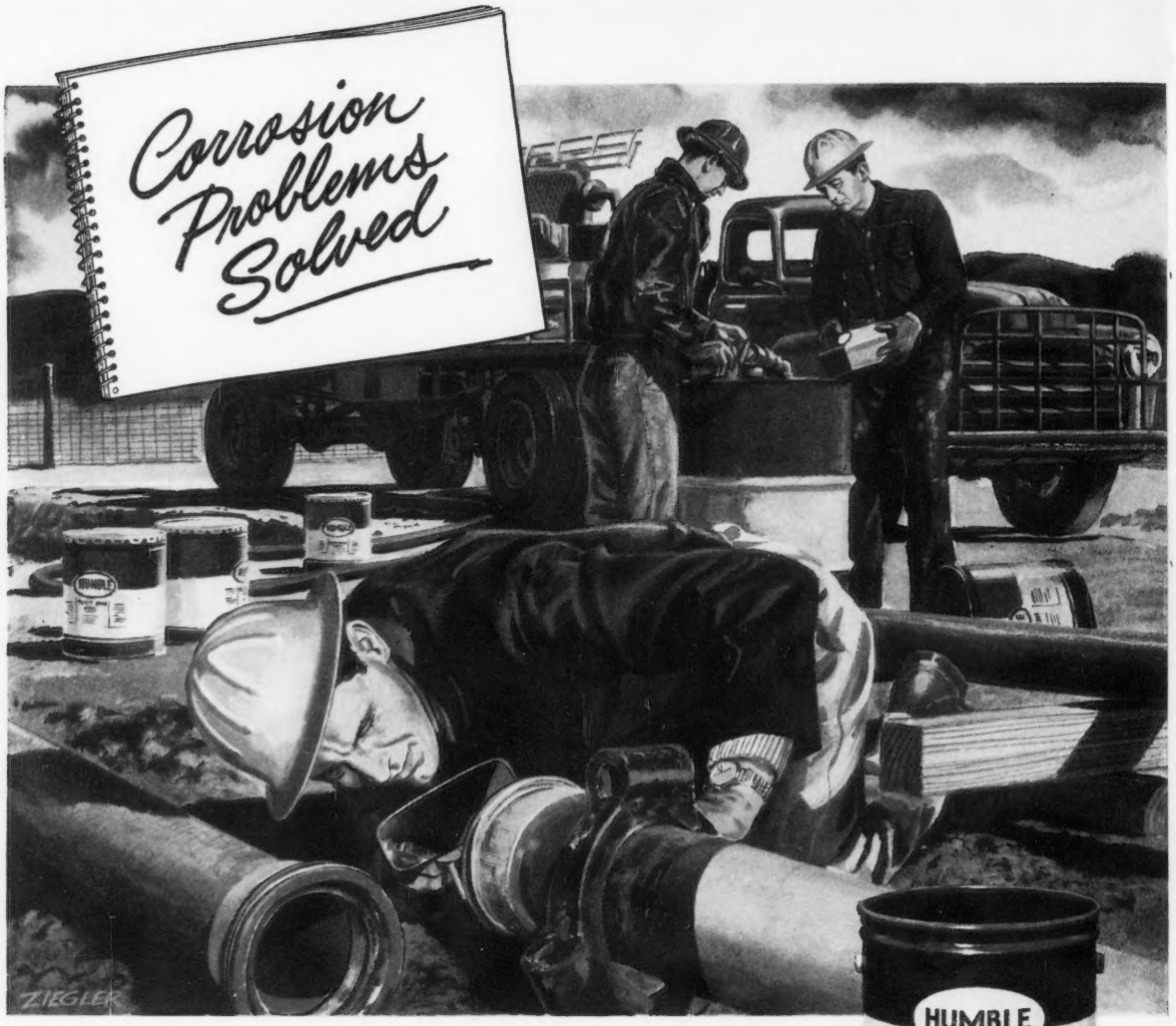

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Humble internal pipe coatings are also available in vinyl and phenolic formulations.

For more information on Humble's complete line of protective coatings, see your nearest Humble wholesale plant in Texas and New Mexico, or write or phone:

HUMBLE OIL & REFINING COMPANY

HUMBLE

Technical Service
Sales Department
Humble Oil & Refining Company
P. O. Box 2180
Houston 1, Texas

HERE'S HOW **DURIRON**®
ended yearly anode replacement

INSPECTION RESULTS AFTER 440 DAYS' SERVICE

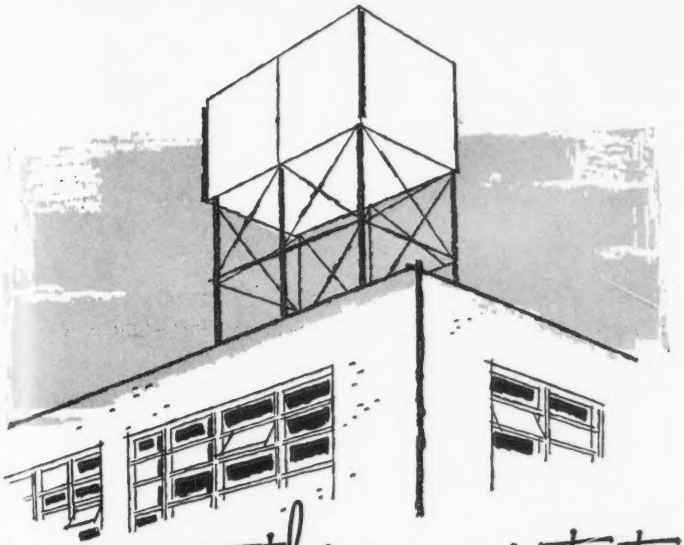
Anode Number	Total Energy Discharge (Ampere Hrs.)	Weight Loss (Ounces)	Consumption Rate Lbs./Amp./Yr.	Current Density (Amps. per Square Foot)
1	3400	1	0.160	0.21 to 0.28
2	3181	½	0.086	0.20 to 0.27
3	3600	2	0.300	0.22 to 0.30
4	3000	¼	0.045	0.17 to 0.23
5	3400	1 ½	0.240	0.22 to 0.30
6*	8700	3	0.190	0.14 to 0.21

**Original test Duriron anode after 751 days in service.*

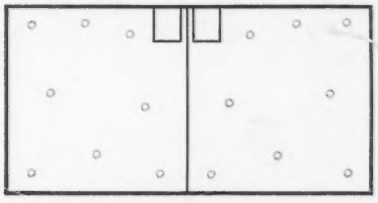
Two 2500 gallon storage tanks had been cathodically protected with aluminum anodes, which had to be replaced annually. In a trial installation, a single DURIRON anode was installed in parallel with fifteen aluminum anodes for a test period of 311 days. At the end of this period, the test was so successful that a complete DURIRON installation was made.

After 440 days of actual service, six of the DURIRON anodes were inspected. The results of this inspection are tabulated above. Anode No. 6 in the table is the original test anode, which at the time of this inspection had been in operation for 751 days. Is the customer satisfied? Sure! Who wouldn't be satisfied with performance like this?

If you use anodes for cathodic protection, DURIRON ANODES will probably do a better job for you. We'd like to prove this to you. Just call or write: THE DURIRON COMPANY, INC., Dayton, Ohio. Telephone: KEnmore 2121.



ARRANGEMENT OF ANODES IN TANKS



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DURIRON®
impressed current
ANODES

THE DURIRON COMPANY, INC., DAYTON, OHIO

The biggest mistake is the one you can't see!

As an experienced pipeline man, you'll have little trouble spotting four obvious "errors" in this picture*. But the *biggest* and most *costly* error you can make in pipeline construction is to gamble on unproven or "economy" types of pipeline protection.

Sure, you can buy "protection" that will save you a few first-cost dollars. But wait until you start paying the bills for excessive cathodic protection!

On line after line, in marshlands and mountains, tough, durable *Pitt Chem* Coal Tar Enamels have *proved* their superior ability to resist soil stress and water absorption, the two pitfalls of "economy" coatings. It's the kind of protection that gives you maximum insurance against high maintenance cost.

When you specify *Pitt Chem* you get assured quality, for *Pitt Chem* Coal Tar Enamels are manufactured to rigid, *published* specifications, from highest grade materials.

How can we help you! Send us the details of your coating problem.

*Operating from left side of ditch; man standing on line; man in ditch under line traveling machine; loop in "Hoosier" pole.

PITT CHEM® TAR BASE ENAMELS

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- Modified Grade
- Plasticized Grade
- Hotline
- Cold Applied Tar Base Coatings

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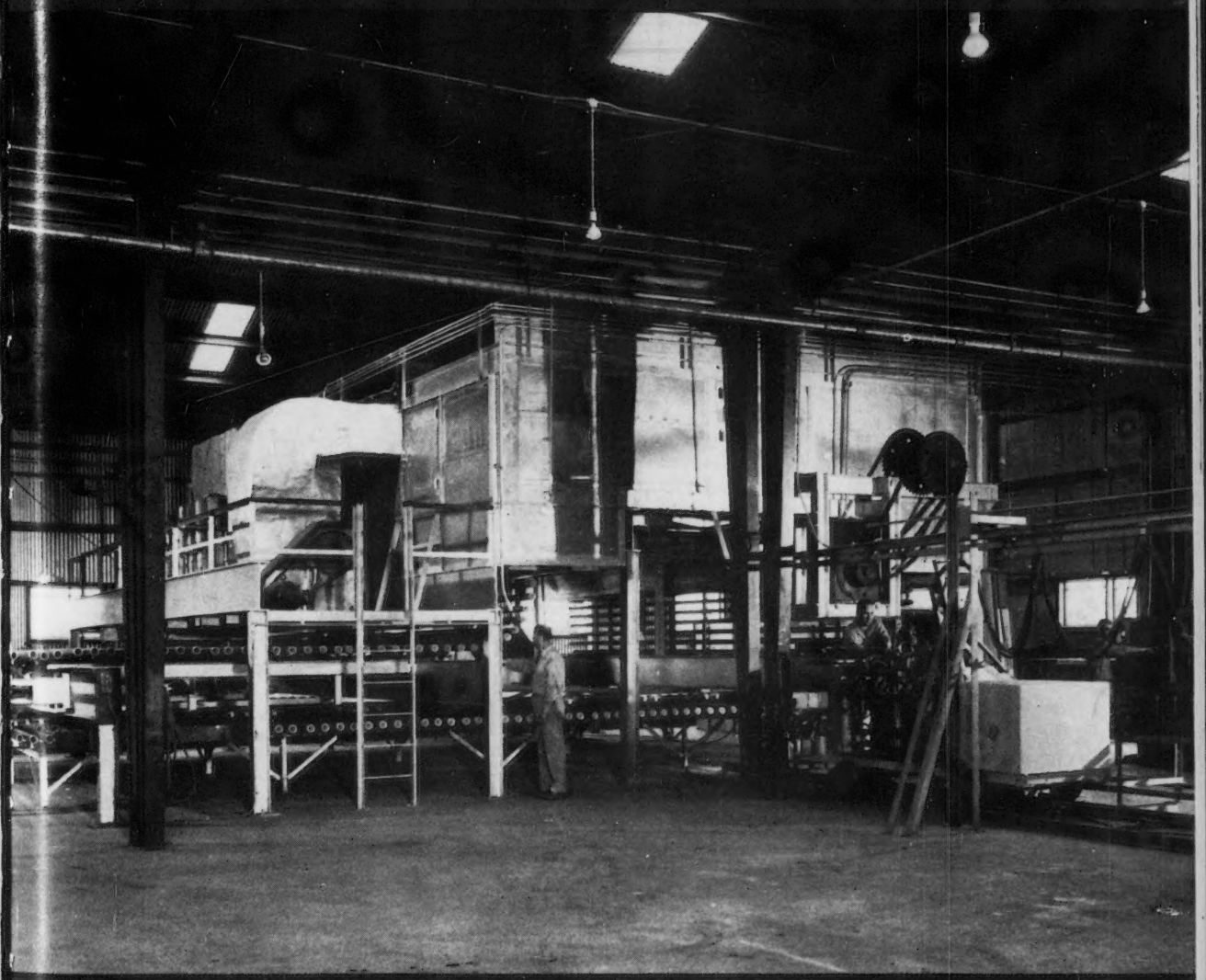

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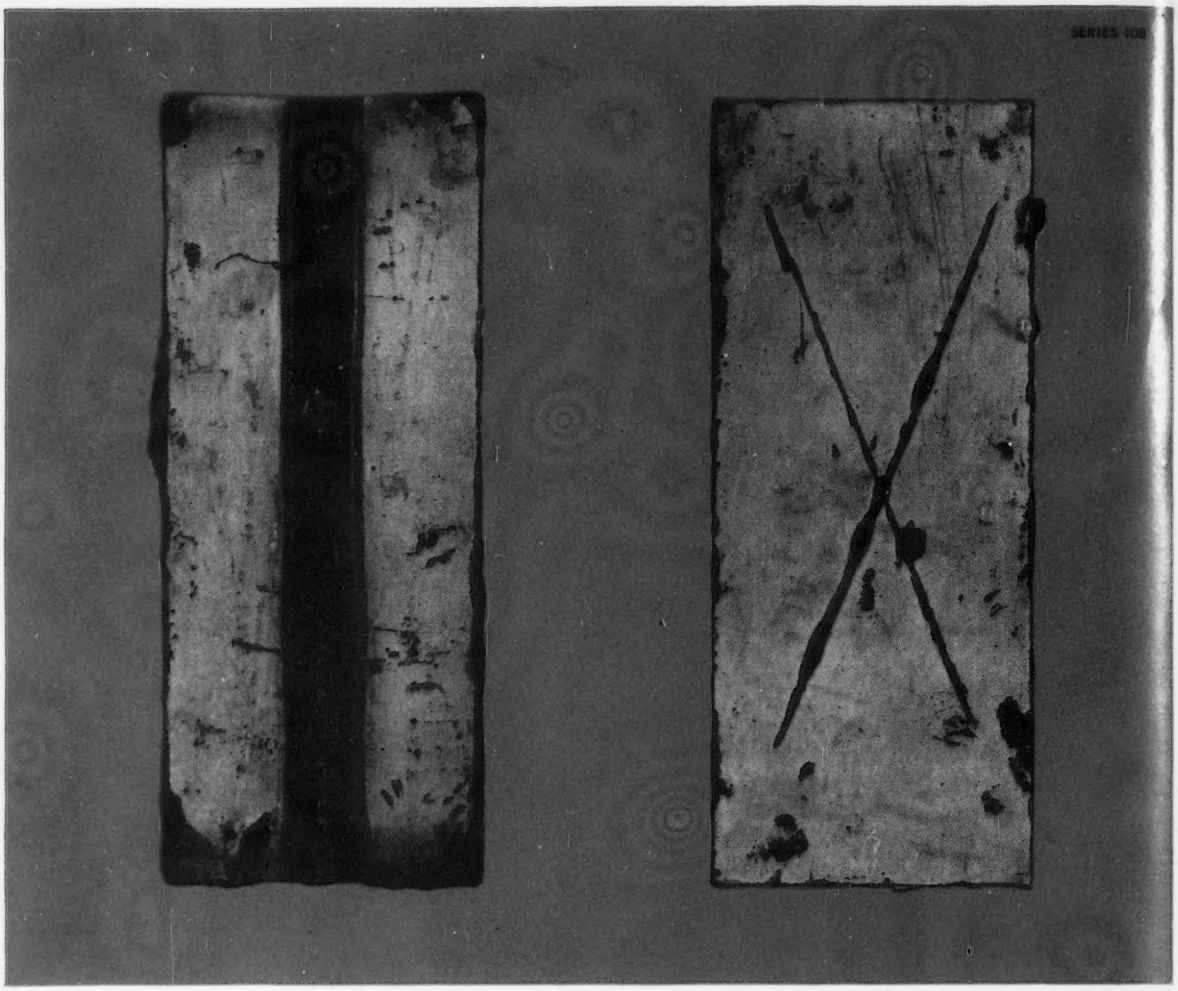

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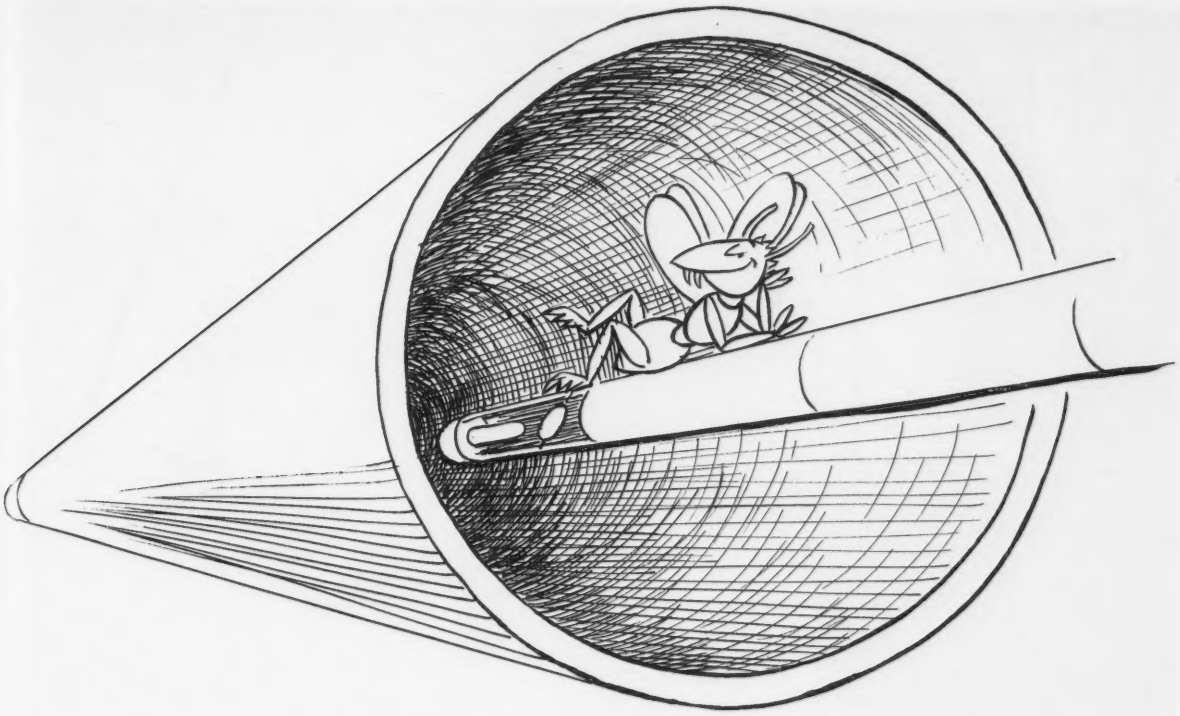

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"These Galvomag anodes are a small investment to protect $20,000 worth of well casings"

Cathodic protection with Galvomag® magnesium anodes is a smart move for any oil well operator.

Corrosion can ruin well casings in a surprisingly short time. Frequently it causes leakage in the casing that floods the producing horizon. Sometimes it causes the casing to collapse. All too often, corrosion results in a costly repair job or abandonment of the well.

A relatively small investment in Galvomag anodes will insure any underground metal structure against corrosion. Galvomag is the name of the Dow high-potential anodes that deliver 25% more current than conventional anodes. In normal soils they give good protection with fewer units. In high-restivity soils they provide the extra current needed to assure good protection.

Contact one of the Dow magnesium anode distributors below for further information and technical assistance, or write to us. THE DOW CHEMICAL COMPANY, Midland, Michigan, Department MA-1427GG1.

Call the distributor nearest you: **Cathodic Protection Service, Houston, Texas • Corrosion Services, Inc., Tulsa, Oklahoma • Electro Rust-Proofing Corp. (Service Division), Belleville, N. J. • Ets-Hokin & Galvan, San Francisco, Calif. • The Harco Corp., Cleveland, Ohio • Royston Laboratories, Inc., Blawnox, Penna. • Stuart Steel Protection Corp., Plainfield, N. J. • The Vanode Co., Pasadena, Calif.**

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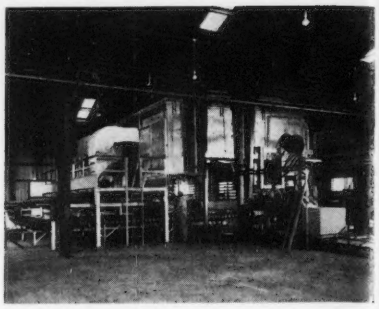

Photo: Tubular Lining Corp.

THIS MONTH'S COVER—Tubing in this machine travels on an endless track through successive cycles of internal coating from the spray head (right) through the baking oven (top) with precise electronic timing to produce a film with optimum uniformity. Phenolics, epoxies or rubber can be applied in successive films to form coatings of required thickness. Consistently homogeneous coatings are produced.

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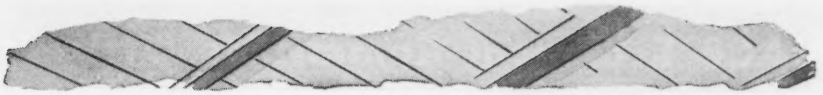

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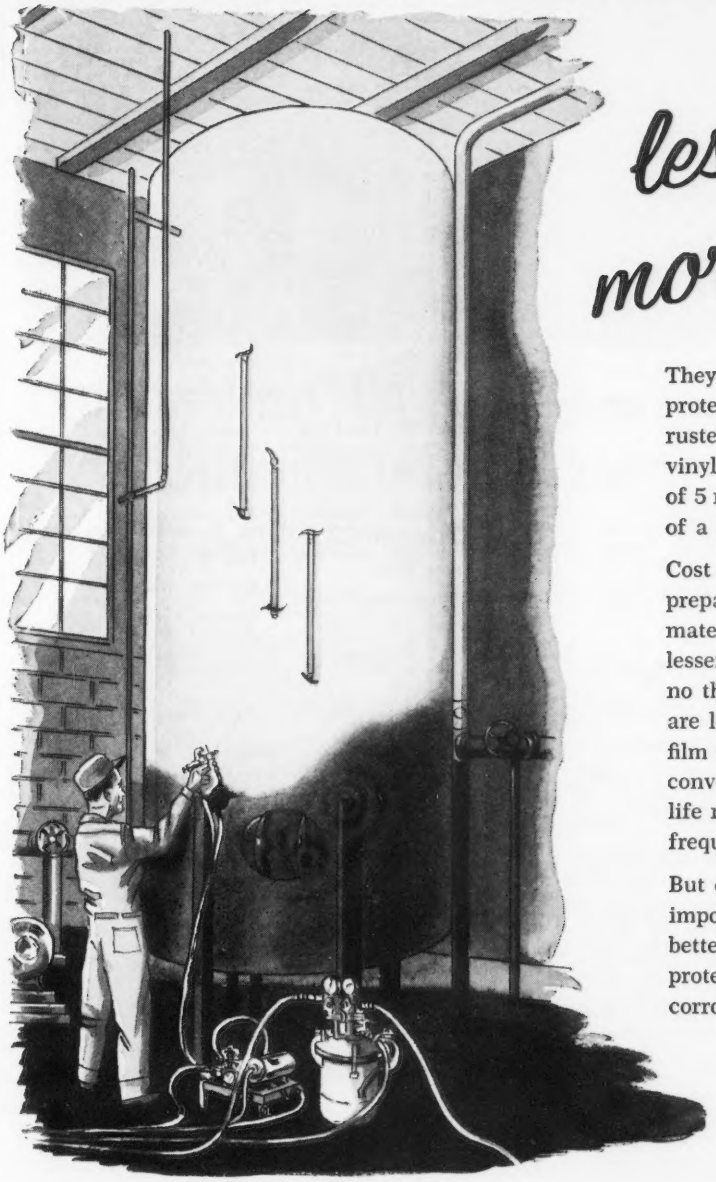

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(Continued on Page 8)

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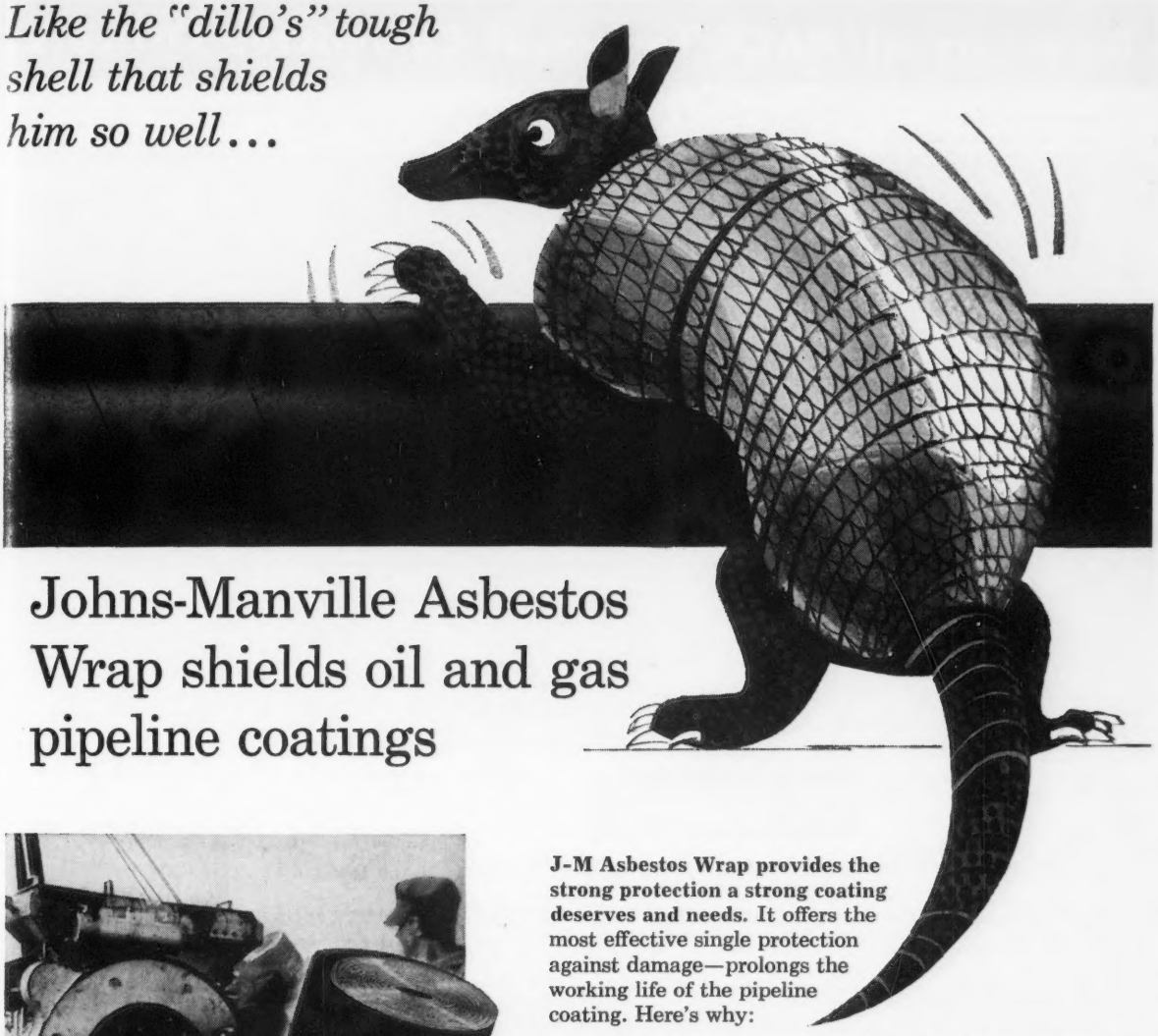

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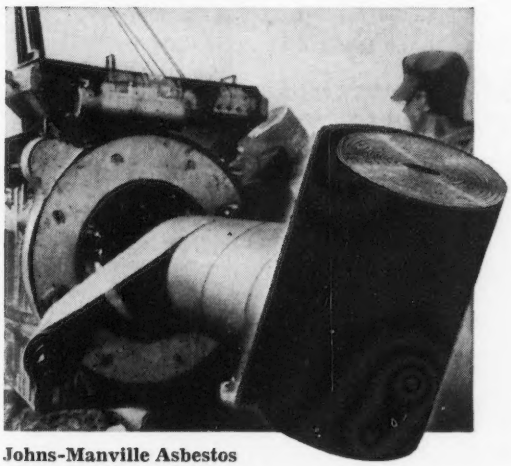

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T-5D-6 Organic Chemicals
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Wade Wolfe, Jr., Secretary, 73 Ermann Drive, Buffalo 17, New York.

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Management says YES

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Let the big G do for you what it did for the men in Tacoma. There's an entire family of GACO heavy-duty corrosion-protection products and services available and a GACO Corrosion Specialist in your area prepared to serve your needs. For complete information *write*—we'll forward literature of interest.

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(Continued on Page 12)

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galvanized iron can now be painted *without* weathering

Time was when galvanized iron downspouts like this one had to weather at least six months before they could be painted. Not any more! Now the painter can prepare new galvanized iron for immediate top coating. With application of a wash primer based on Shawinigan's FORMVAR or BUTVAR, painting can be done half an hour after priming.

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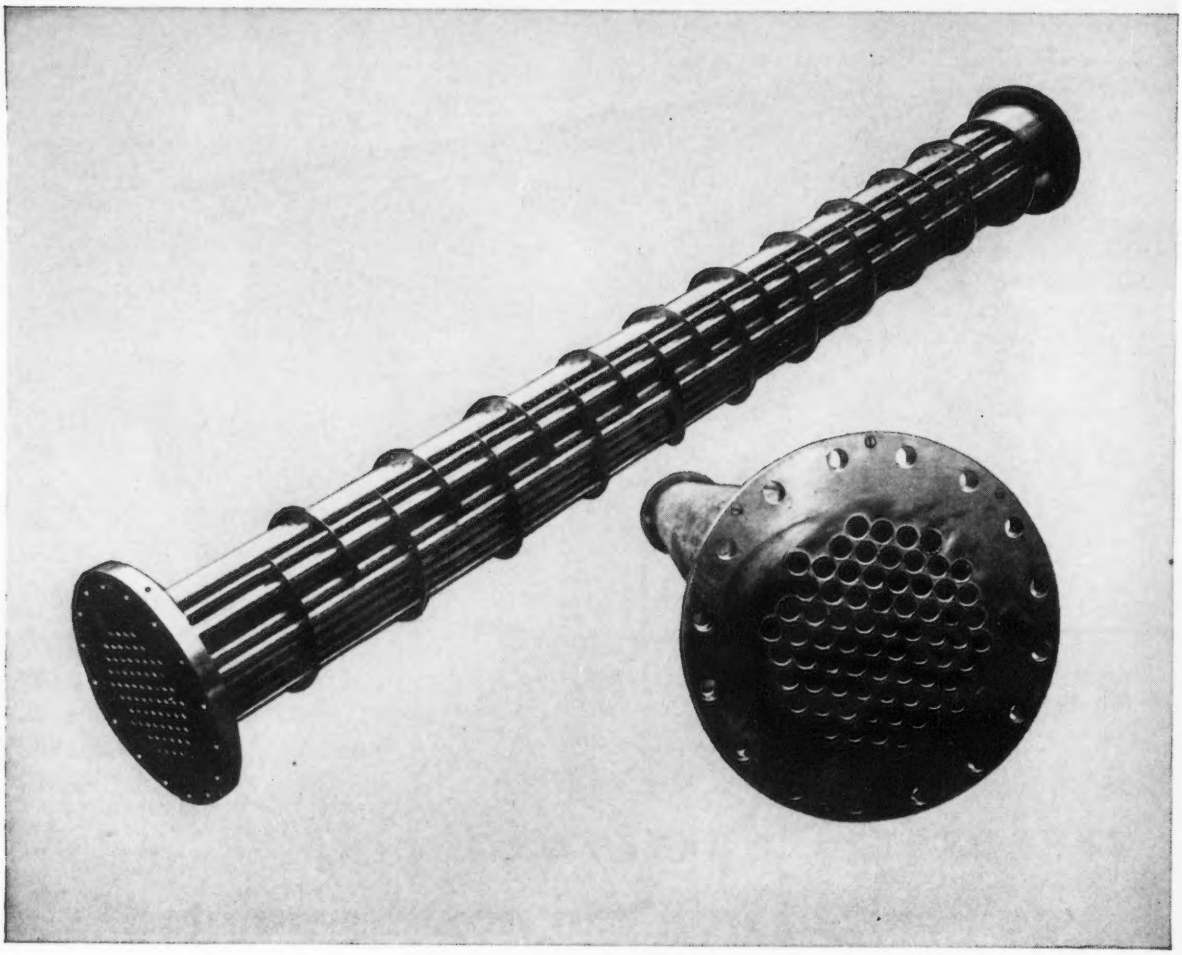

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Titanium condenser tubes expected to outlast stainless 30 to 1 handling nitric acid

Corrosive vapors of 60% nitric acid gnawed through an average of three stainless-steel tube bundles a year in a chemical firm's condenser. Maintenance and replacement costs were running as high as $18,000 a year. Then titanium tubes were installed. Based on a service life of 10 years, titanium will save at least $162,000 in maintenance and replacement costs.

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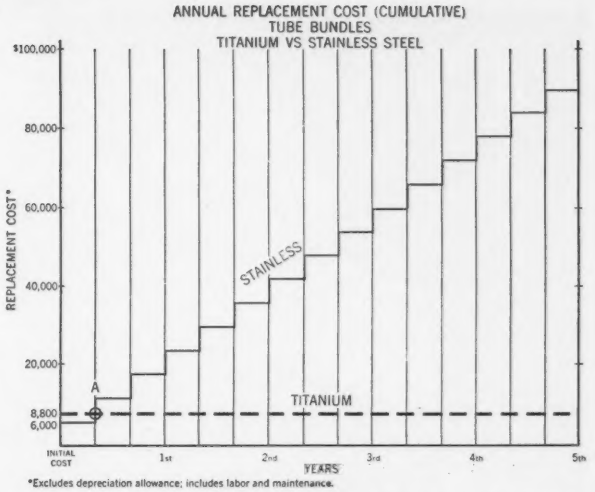

INITIAL AND ANNUAL REPLACEMENT COSTS of titanium and stainless tube bundles are compared above. Initial cost of the titanium tube bundle, shown at left, is $8,800, vs. $6,000 for stainless. But note how the $2,800 difference is absorbed within 4 months of service (Point A). Savings are approximately $81,000 after 5 years . . . $162,000 after 10 years, the expected service life of titanium tubes.

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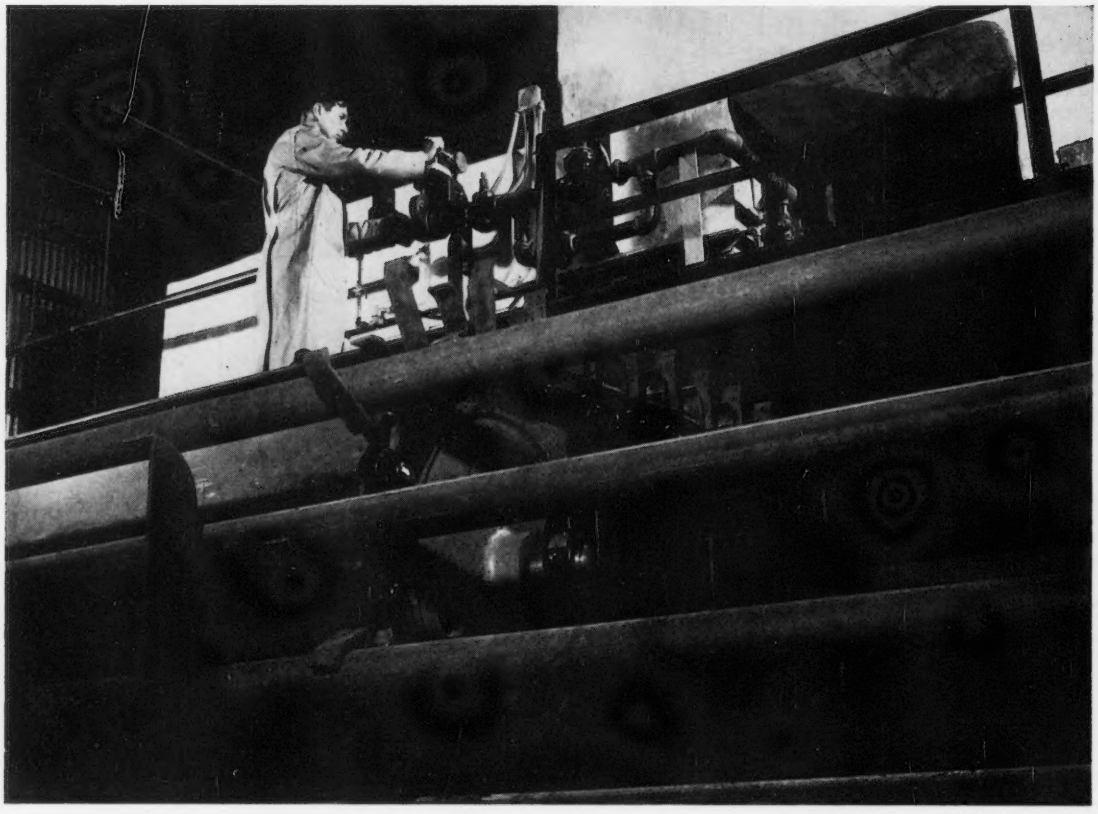

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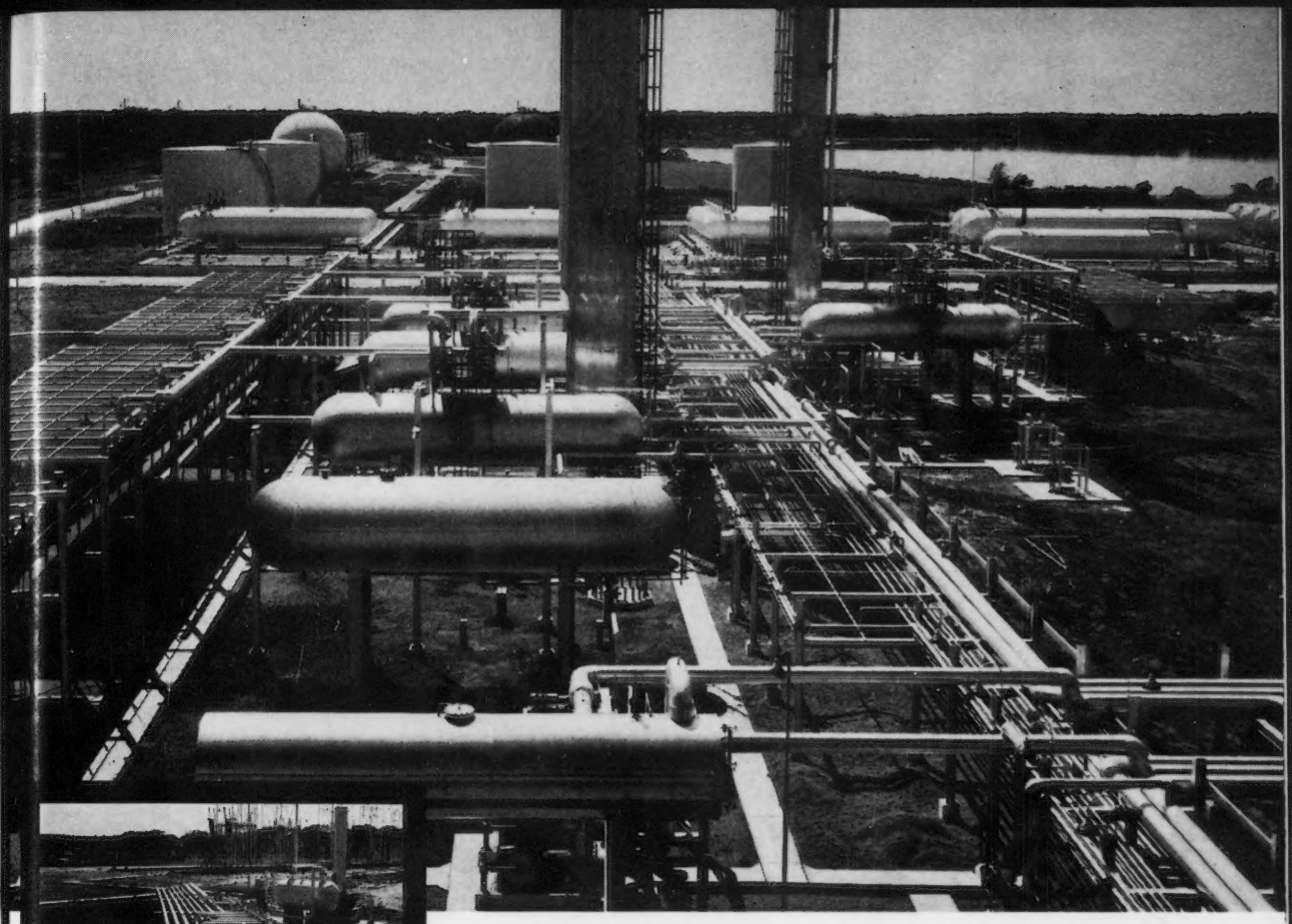
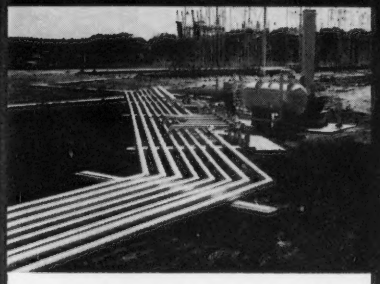


Three-coat systems of Normal Copon self-cleaning white tank coating and three-coat systems of Normal Copon aluminum tank finish were used to protect this fractionating plant of The Bay Petroleum Corp. (a subsidiary of Tennessee Gas Transmission Co.).



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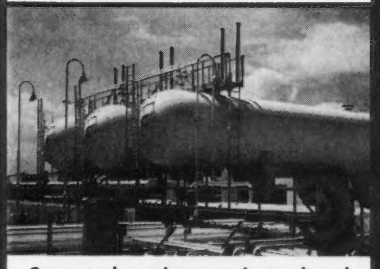

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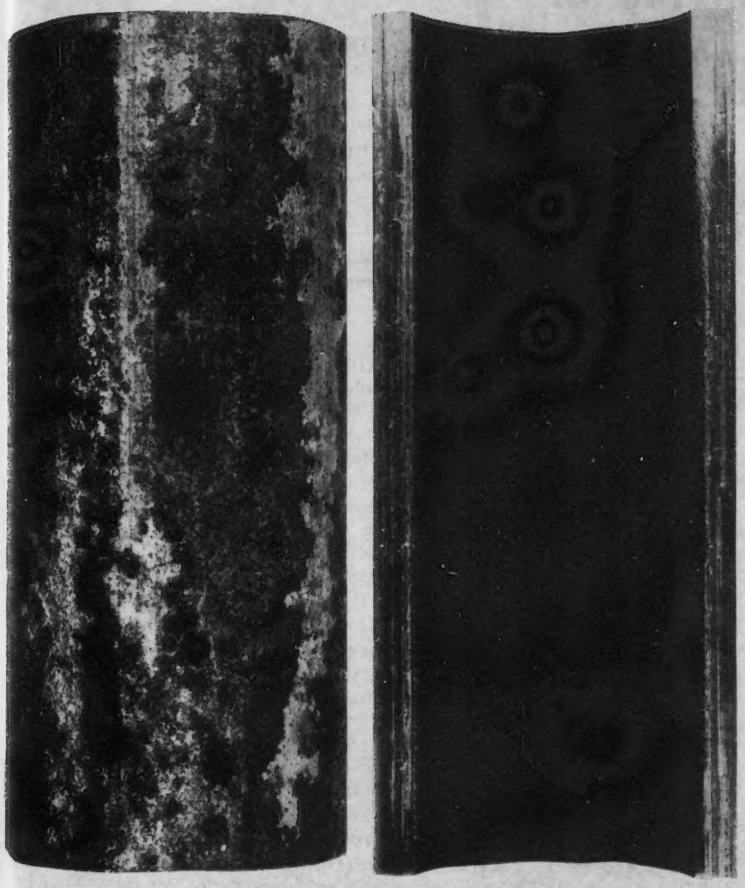

65
YEARS OF SERVICE

Longitudinal photomicrograph U-2-3 (100x) shows a typical area—parallel to rolling—of the microstructure observed in the subject 1¼" wrought iron pipe.

Metallurgical Report 1517—Condensate Return Line*

This report deals with the metallurgical examination of a length of 1¼" wrought iron pipe, submitted to our laboratory for investigation and comment.

The outer surface had a tightly adherent oxide coating and was found to be in excellent condition. A 4-inch long piece of the sample was split longitudinally into two half-sections. The interior surface was covered with a rust film; no localized pitting or grooving was observed.

Chemical composition of the pipe sample is as follows: Carbon—.027%; Manganese—.050%; Phosphorus—.217%; Sulphur—.026%; Silicon—.150%; Iron Silicate—3.98%.

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(a) To promote the prevention of corrosion, thereby curtailing economic waste and conserving natural resources.

(a) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

(d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.

(e) To promote standardization of terminology, techniques, equipment and design in corrosion control.

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Corrosion

OFFICIAL MONTHLY PUBLICATION

National Association of Corrosion Engineers

An Association of Individuals and Organizations Concerned with Control of Corrosion

1061 M & M Building Houston 2, Texas

Vol. 13 September, 1957 No. 9

Topic of the Month

Coating Resistance Conforms to Probability Law

By F. E. KULMAN*

Introduction

COATING RESISTANCE measurements on buried pipes are made for several reasons including (1) the determination of cathodic protection requirements, (2) the rate of deterioration of the coating, and (3) the maintenance of the pipe free of accidental contact with other buried structures.

As test data on coatings accumulated it was found that coating resistance depends on the quality of construction, resistivity of the soil, the duration of burial, the season of the year, soil temperature, and possibly other factors. Statistical techniques have been found to be very helpful in analyzing and interpreting the test results. This article describes such a study.

In 1947, a 138-kv pipe type cable feeder consisting of a 6-inch steel pipe, 41,535 feet long, containing cables, was installed between the Jamaica substation of the Consolidated Edison Company of New York and the Valley Stream substation of the Long Island Lighting Company. The soil was generally a brown sand with some clay, and had a resistivity ranging from 5,000 to 20,000 ohm-centimeters. At the time of pipe and manhole installations, sections of spare pipe were installed for future use when a second feeder would be required.

During the period 1947 to 1955, coating resistance measurements were made on the 17 isolated pipe sections in the Consolidated Edison Company portion of the feeder route. These measurements are tabulated in Table 1. In 1957, cables were installed in the spare pipe sections and spliced to provide the second feeder. The pipe sections were then joined together at the manholes.

Distribution of Soil Resistivity Measurements

A satisfactory method of analyzing the accumulated test data was lacking until Dr. Gordon N. Scott presented his paper[1] on the distribution of soil resistivity measurements. This paper showed that the distribution of the logarithms of soil resistivity measurements along a particular pipe line usually conformed to the normal probability curve, and that plotting the measurements on log-probability paper resulted in a linear plot.

Dr. Scott's technique was adapted to the analysis of the coating resistance measurements of Table 1 and the results are shown in Figure 1. The scale of ordinates is a logarithmic one and the abscissa scale is distorted in such a manner that the integral of a normal probability curve plots as an inclined straight line. Log-probability paper is available from suppliers of drafting equipment.

To plot the distribution of measurements for a given survey, the resistance data are arranged in order of increasing magnitude. The probability asso-

TABLE 1—Coating Resistance Measurements on 17 Pipe Sections of Feeder 902

SECTION			COATING RESISTANCE—MEGOHMS PER SQUARE FOOT				
From MH	To MH	Length Feet	Sept. 1947	Mar. 1950	Jan. 1951	Feb. 1953	May 1955
7001	7002	564	68.	14.3	3.86	0.598	0.034
7002	7003	722	510.	26.8	1.14	1.10	0.253
7003	7004	1,983	190.	22.4	1.49	0.654	0.249
7004	7005	1,300	527.	24.3	7.45	1.06	0.157
7005	7006	1,375	47.	21.0	4.87	0.971	0.209
7006	7007	1,963	11.1	10.8	3.10	1.35	0.392
7007	7008	2,053	5,890.	94.2	4.64	1.61	0.399
7008	7009	2,047	1,010.	37.4	2.17	0.483	0.000
7009	7010	2,002	194.	21.0	3.30	1.38	0.341
7010	7011	1,991	169.	28.9	6.41	1.28	0.388
7011	7012	2,038	64.	34.4	5.71	1.05	0.278
7012	7013	2,076	169.	7.54	5.57	0.846	0.276
7013	7014	1,891	228.	33.8	2.40	0.861	0.258
7014	7015	1,964	336.	52.2	11.3	1.48	0.333
7015	7016	1,877	538.	17.6	5.04	0.678	0.268
7016	7017	1,740	815.	8.5	4.10	1.23	0.314
7017	7018	1,514	107.	22.9	4.06	1.97	0.397

TABLE 2—Coating Resistance Values

DATE	MEGOHMS PER SQUARE FOOT		
	5% Level	50% Level	95% Level
September, 1947	28.5	230.	1900.
March, 1950	6.7	24.5	83.
January, 1951	1.35	4.0	12.
February, 1953	0.48	1.05	2.2
May, 1955	0.19	0.285	0.43

* Consolidated Edison Co. of New York, Inc., New York, N. Y.

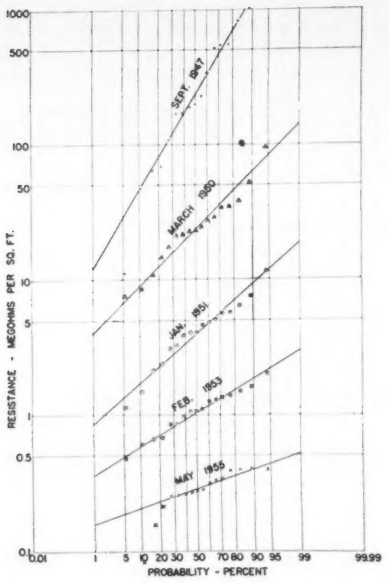

Figure 1—Coating resistance of 17 pipe sections of feeder 902 pipe.

ciated with any measurement is calculated by dividing the rank of that resistance measurement by a number equal to the number of measurements plus one. Thus, the computed probabilities of the individual values of the 17 measurements are 1/18, 2/18, 3/18 . . . . . 17/18, equal to 5.5 percent, 11 percent, 16.5 percent . . . . . 94.5 percent respectively.

Reference to Figure 1 shows that in general the coating resistance measurements plot as straight lines, indicating conformance to the normal probability curve.

The median coating resistances of the 17 sections decreased with time and there was a roughly proportional decrease in the range of variation about the median value as shown by the slopes of the lines. The coating resistance values at 5 percent, 50 percent (median) and 95 percent levels are shown in Table 2.

The rate of decrease of the median was in the order of 60 percent per year. The reasons for the decrease have not been completely determined but apparently are related to the absorption of soil moisture by the coating. After eight years, the coating resistance was still good, although changing.

If a coating resistance value does not fall on or near the line, it is an indication of a disturbing factor. This may result from an error in the measurement, an accidental contact between the pipe and another metallic structure, or some other cause which can be investigated and isolated. For example, the 1955 tests showed an extremely low resistance in one section, less than 0.1 ohm. Excavation of the section at the suspected point of low resistance revealed the coated pipe to be in metal to metal contact with another pipe. When the contact was cleared, the resistance became 75 ohms, equivalent to 0.23 megohm per square foot.

The use of the log-probability paper facilitates the interpretation of coating resistance measurements and wider application of the method is foreseen.

Reference

1. Gordon N. Scott. The Distribution of Soil Conductivities and Some Consequences. Paper No. 44 presented at Thirteenth Annual Conference, NACE, St. Louis, Mo., March 11-15, 1957.

DISCUSSIONS ON TECHNICAL ARTICLES ARE SOLICITED

Discussions on technical articles published in Corrosion will be accepted for review without invitation. Discussions must be constructive, accompanied by full substantiation of fact in the form of tables, graphs or other representative data and be submitted in three typewritten copies.

Authors of discussions are asked to supply one copy of figures suitable for reproduction and will be sent on request a copy of the NACE Outline for the Preparation and Presentation of Papers.

Discussions will be reviewed by the editor of Corrosion and will be sent to the author of the paper discussed for his replies, if any. Publication will be in the Technical Section with full credit to the authors together with replies. Discussions to papers presented at meetings of the association may be submitted in writing at the time of presentation or later by mail to the editorial offices of Corrosion, 1061 M & M Bldg., Houston 2, Texas.

Resistance to Corrosion of Aluminum Alloys For Automotive Applications*

By E. T. ENGLEHART, W. C. COCHRAN and E. P. WHITE

Introduction

THE USE of aluminum alloys in automotive applications is almost as old as the automotive industry itself. In fact, early history of automobiles shows that aluminum was one of the major components employed in their construction. In the early 1920's, some automobiles used aluminum alloys to the extent of 35 percent of the total weight of the car. However, with the increase of mass production methods and the advent of less expensive, deep-drawing steels, the use of aluminum alloys in automotive applications declined. After World War II, aluminum alloys again found increased use in many functional parts such as pistons, clutch housings, timing and distributor gears and cylinder heads. Carburetor and automatic transmission parts also appeared in aluminum. Recently, because of the remarkable development of attractive finishes and corrosion-resistant alloys, interest in aluminum body and trim members has grown sharply. The purpose of this paper is to discuss this anticipated use of aluminum.

In addition to laboratory test data, the good service performance of aluminum alloys as automotive trim has been substantiated by atmospheric exposure tests. One manufacturer has employed extruded aluminum window frames on production model cars for six years with excellent results. Another manufacturer has used anodically coated aluminum trim in several areas in production models for the past few years. These trim members have provided excellent performance. Even longer service has been obtained on experimental aluminum parts on a car tested by Alcoa. This car employed aluminum for many body and trim parts with equally satisfactory results. These materials, which will be discused in detail, also indicate that aluminum alloys should provide good performance in this application.

Choice of Alloy

There is a wide range of aluminum alloys available today for automotive and other types of applications. These alloys provide a range of mechanical properties, high resistance to corrosion and a variety of surface finishes. Since this paper is directed primarily toward automotive trim applications, only those alloys used for this purpose will be discussed in detail. However, other alloys will be discussed in

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E. T. ENGLEHART—Since graduating from Pennsylvania State University in 1943 with a BS in chemistry, Mr. Englehart has been employed as a Research Engineer in the Chemical Metallurgy Division of the Alcoa Research Laboratories, New Kensington, Pa. His work has been associated with fundamental and practical problems of corrosion of aluminum alloys as well as the study of methods of preventing corrosion. Mr. Englehart is a member of NACE.

W. C. COCHRAN—After graduating from Carnegie Institute of Technology in 1942 with a BS in chemical engineering, Mr. Cochran served for three years in the U. S. Army Corps of Engineers. In 1946 he joined Alcoa Research Laboratories as a Research Engineer. He has been engaged in research and development work concerning surface finishes for aluminum, specializing in decorative and protective anodic coatings and coloring and brightening methods. Mr. Cochran is a member of NACE and Electrochemical Society.

E. P. WHITE graduated from Cornell University School of Civil Engineering in 1941 and was employed until 1944 by American Magnesium Corp., Buffalo, N. Y. In 1945, after eighteen months service with the U. S. Army Air Corps, he joined Alcoa, specializing in structural and mining projects. In 1949 he was made Head of the Automotive Section, the position which he now holds. Mr. White is a member of Society of Automotive Engineers.

Abstract

Aluminum alloys have been employed in automobiles for some time in applications such as pistons. In recent years, however, increased use of aluminum alloys for many other automotive applications has been anticipated. This paper presents the available information on the resistance to corrosion of aluminum alloys for these many new uses. Particular emphasis has been placed on performance of aluminum alloys for automotive trim, but other applications are also covered. Included is a discussion of choice of alloy and the performance of the large variety of finishes available. Results of atmospheric corrosion tests together with those of limited field experience are provided to indicate the good resistance to corrosion of aluminum alloys in these applications. Factors affecting the performance of these alloys in service are also covered.

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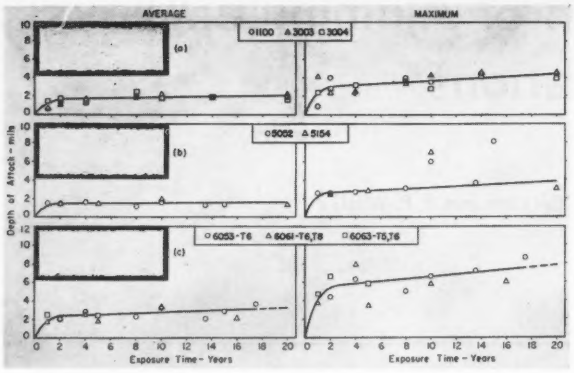

Figure 1—Curves showing the average and maximum depth of attack of various unprotected aluminum alloys after exposure of as long as 20 years to the industrial atmosphere at New Kensington, Pennsylvania. The inserted micrographs of typical cross sections show the extent of corrosion encountered on each class of alloy after exposure of 20 years to this environment.

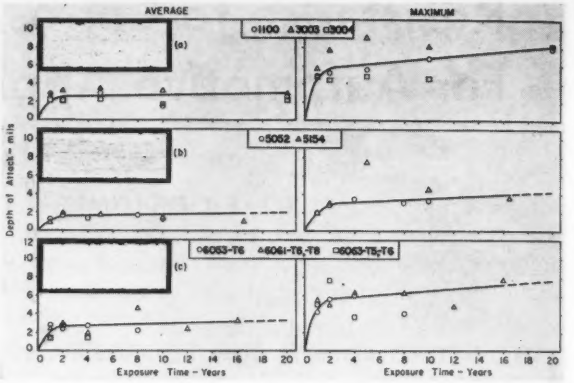

Figure 2—Curves showing the average and maximum depth of attack of various unprotected aluminum alloys after exposure of as long as 20 years to the seacoast atmosphere at Point Judith, Rhode Island. The inserted micrographs of typical cross sections show the extent of corrosion encountered on each class of alloy after exposure of 20 years to this environment.

general to help in establishing the performance of the trim alloys.

One alloy employed for trim application is 3003. This alloy contains approximately 1.25 percent manganese. It is recommended that 3003 alloy be employed where a diffuse finish is desired.

In applications in which trim of high specularity and reflectance is required, alloy 5357 is most commonly used. This alloy contains approximately 1 percent magnesium and 0.3 percent manganese and is of somewhat higher purity than 3003 alloy. It provides a pleasing, bright finish that has a high resistance to corrosion.

These alloys will maintain their structural integrity in very corrosive atmospheres over an extremely long period of time without protection. Exposure of 20 years to the seacoast atmosphere at Point Judith, Rhode Island, and to the industrial atmosphere at New Kensington, Pennsylvania, has indicated that the change in tensile strength and average depth of attack caused by corrosion were of a very small magnitude for alloys of this type. Application of protective and decorative coatings to these alloys permits the easy maintenance of a good appearance. This improvement is of great importance in automotive trim applications since it is imperative that automotive trim maintain good appearance (by virtue of the absence of surface effects), as well as its structural integrity.

The effect of impurities in aluminum in reducing the specularity and brightness obtainable with anodic oxide finishes is well known. By using very high purity aluminum, it is possible to produce alloys which, after anodic oxidation, will have a specularity and brilliance superior to that obtained with the commonly employed commercial alloys. Aluminum alloys of very high purity are currently being used for automotive trim in Europe and to a limited extent in the United States. With these high purity alloys, thicker and more protective anodic coatings can be applied without appreciably reducing the specularity and brightness.

The price of these high purity alloys limits their use in this country at the present time. It is possible, however, that alloys of intermediate purity will provide a more satisfactory appearance than presently used commercial alloys and still be economical for general automotive trim applications.

Finishes

One of the advantages of using aluminum in any application is the wide variety of finishes, both decorative and protective, that can be applied to it. In this respect, aluminum, including those alloys used for automotive trim, is probably more versatile than any other commonly employed metal. Not only can the metal be used satisfactorily in its as-fabricated state for many applications but also the many different finishes that are available will usually improve the performance of the material. Aluminum alloys can be finished mechanically, by chemical methods, by electrochemical methods, and by the use of paint, lacquers or enamels.

In the use of aluminum alloys for automotive trim applications, anodic oxidation and electroplating treatments are most commonly employed. Although these will be discussed in some detail, it should be pointed out that the anodically coated samples as discussed in this paper were coated by the Alumilite process. This is pointed out since there are several methods of application of anodic coatings that vary in some respects.

Some aluminum alloy components, such as scuff plates, are successfully used without the application of a protective coating. Where it is desirable to maintain a decorative appearance, however, an anodic oxidation treatment is usually recommended. The use of an anodic oxide coating on aluminum alloys improves not only the resistance to weathering but also the resistance to abrasion of the surface of the aluminum member. In general, resistance to weathering and abrasion improves with increased thickness of anodic coating. In other instances, chromium plating has been used for finish on forged wheels, permanent mold cast handles, bumper ends and other similar parts. In these cases, high resistance to scuffing and abrasion is obtained along with satisfactory resistance to corrosion.

In most cases where anodic finishes are to be applied to aluminum alloys, a procedure such as the Alumilite process is used. This process employs a dilute sulfuric acid solution as the electrolyte and provides coating thicknesses roughly proportional to the time of coating application.[1,2] For automotive trim applications, a treatment of 10 to 30 minutes duration (0.00015 to 0.00045 inch coating thickness) is usually adequate. For interior trim applications in which the environment is relatively non-corrosive, anodic treatments of as short a duration as 10 minutes (0.00015 inch) can be employed, while treatments of at least 15 minutes duration (0.00023 inch) are usually used to resist the weathering effects encountered in exterior applications. Of course, the thickness of coating considered necessary for these applications may change to some degree with further service experience. In this respect, it might be pointed out that there is a difference in the thickness requirements of anodic coatings for automotive trim applications as compared to those for architectural applications. Architectural applications of anodically coated aluminum alloys are usually expected to last 30 to 40 years or more with only infrequent maintenance. In contrast, the life of an automobile is relatively short and regular maintenance is an almost accepted procedure. As a result, the anodic coatings employed in architectural applications must be of a greater thickness than those employed in trim for satisfactory performance. Anodic treatments of at least 30 minutes duration and in most cases 45 to 60 minutes are employed in exterior architectural applications.

After the application of the coating to aluminum members, a sealing operation in clean boiling water is carried out. It is important that this operation be done properly to obtain the best resistance to corrosion and staining. Color can be applied to the anodically coated part by impregnating the oxide coating with a colorant prior to sealing. At the present time, gold finishes have been applied to exterior aluminum automotive trim members by this method and are being used successfully by the industry. Other colorants are now in the process of development for this application and should be available in the near future.

Electroplating is a commercially practical and economically sound method for finishing aluminum for many applications. This process will provide a pleasing finish with good wear resistance. For decorative finishes, electroplates of chromium, nickel, copper, brass, silver and gold have been used. For chromium plating on aluminum, the zinc immersion process is the most generally applicable method of surface preparation and is satisfactory for use with practically all of the commercial aluminum alloy products.

Resistance to Corrosion

Over the past 30 years, Alcoa Research Laboratories have conducted many tests to determine the resistance to corrosion of aluminum alloys either in the unprotected state or when protected by the large variety of finishes available. In general, these corrosion tests include exposure of samples to conventional salt spray equipment, the seacoast atmosphere at Point Judith, Rhode Island, and to the industrial atmosphere at New Kensington, Pennsylvania.

The exposure station at Point Judith, Rhode Island, provides a very severe seacoast environment since it is located only 300 feet from the ocean. The coast is rocky and the sea often rough in this location which means that considerable salt mist is usually present in the air.

The industrial atmosphere test station is located on the roof of Alcoa Research Laboratories at New Kensington, Pennsylvania. That city is highly industrialized without smoke control. In addition the roof of the laboratory contains several chemical exhaust hoods which make the environment more industrialized than those atmospheres normally encountered.

The resistance to corrosion of aluminum alloys is usually evaluated by visual inspection, microscopic examination for the depth of penetration of corrosion and by determination of the change in tensile strength of the alloy as a result of corrosion. Extensive tests have been carried out on the inherent resistance to corrosion of all alloy types.[3] Figures 1 and 2 illustrate the results of microscopic examination on alloys of the same general type used for trim after exposure of 20 years to the seacoast and industrial atmosphere test sites.

Of primary interest in these curves is the illustration of the self-limiting nature of the corrosion which develops on aluminum alloys. In the case of Figure 1, the average depth of attack of the aluminum alloys is approximately 2 mils after two years and only about 2½ mils after 20 years' exposure to the industrial atmosphere. Figure 2, illustrating the results of exposure to the seacoast atmosphere, provides the same general information. Tensile test results from similar specimens show the same trends.

Maintenance of a pleasing appearance is probably the most important quality necessary for automotive trim. Protective methods that will maintain not only the structural integrity but also the good appearance of aluminum alloys are available. Data and illustrations showing the performance of anodically coated samples of aluminum after exposure of as long as five years in various environments have been obtained. Since 5357 alloy is relatively new, it is not possible to illustrate the condition of this material under all these test conditions. Past experience has shown, however, that the resistance to corrosion of 5357 alloy is at least as good as, if not better than, that of the highly resistant alloys illustrated. Consequently, results at least as favorable as those illustrated should be obtained from tests on 5357 alloy.

Samples of 1100 alloy that had been chemically brightened and given anodic oxide treatments of 10 and 20 minutes duration were exposed for four years to the industrial atmosphere at New Kensington, Pennsylvania. These samples were not maintained in any way during the exposure period. Although the sample treated for 20 minutes was superior to that treated for 10 minutes, both have shown a high resistance to corrosion and a pleasing appearance

Figure 3—Photograph showing a 1942 production model car in which aluminum alloy body and trim parts were substituted for the conventional materials by Alcoa. Included among the various aluminum alloy parts were window moldings, radiator grille louvers, windshield moldings, fender moldings, wheels, bumpers, trunk lid, door panels, hood and other small parts. When dismantled in 1955, the aluminum alloy parts revealed performance superior to that of the conventional materials.

at the completion of the exposure period. A similar sample coated for 10 minutes also maintained a pleasing appearance after an exposure of five years to the industrial atmosphere.

In another test, samples of high purity aluminum (alloy 1187) coated for 8 and 16 minutes were exposed for three years to this industrial environment. These samples exhibited a high resistance to corrosion and good appearance although the sample with the 16 minute coating was superior to that employing the 8 minute coating. Similarly, good performance has been obtained from samples such as this after exposure in conventional salt spray equipment.

Samples of 5052 alloy with 10-, 20- and 30-minute coatings were exposed for six months to 20 percent continuous salt spray at 95 F. It was found that while all samples remained in relatively good condition, the samples with the 10- minute coatings were not quite as good as those with the 20- and 30- minute coatings. In all cases, it has been noted that the resistance to corrosion and appearance of anodically coated samples after exposure to these various environments improves with increased thickness of anodic coating.

A number of tests have been conducted in which anodic treatments of five minutes duration have been applied to the aluminum samples. While good results were obtained on these materials in some of the exposure tests, the difference in performance of these materials and those to which 15- and 20- minute coatings had been applied was marked. Thus, it would appear that anodic treatment of at least 10 minutes duration for interior applications and of at least 15 minutes duration for exterior applications should provide satisfactory performance as automotive trim. While treatments of less than 15 minutes duration have provided oxide coatings that have performed satisfactorily in some applications and under some conditions, it is felt that the resistance to corrosion, resistance to surface abrasion and retention of good surface appearance of such coatings may not be as satisfactory as that of the thicker coatings for exterior applications.

A more recent test involved the exposure of anodically coated samples of 5357 alloy with a bright finish. The anodic treatments employed for these samples were of 10, 15, 20 and 30 minutes duration. After exposure of three months to 3½ percent NaCl intermittent spray, it was found that all of the anodically coated panels had remained in remarkably good condition. There was some evidence of minor coating breakdown on the samples with the thinner coatings, particularly the 10- minute coating. However, the breakdowns which had occurred had little effect on the appearance of the materials. Duplicate sets of panels exposed to the industrial atmosphere at New Kensington, Pennsylvania, were in excellent condition with no evidence of breakdown after exposure of three months.

Limited tests to determine the resistance to corrosion of chromium plated aluminum alloys have also been conducted. Panels of 1100 alloy and 2024 (an aluminum-copper type) alloy were plated with approximately 0.0015 inch nickel and 0.00002 inch chromium. One set of panels was exposed to 20 percent NaCl continuous spray at 95 F for 740 hours and a duplicate set was exposed for five years to the industrial atmosphere at New Kensington, Pennsylvania. At the conclusion of these periods of exposure, it was found that the chromium plated aluminum alloys had provided satisfactory performance.

Factors Affecting Corrosion

The corrosion of aluminum alloys as well as any other type of commonly used metal is dependent to a large extent on service and environmental conditions to which the metal is subjected. In environments in which moisture is not present, corrosion is not a problem since it cannot proceed without the presence of an electrolyte. In addition, moisture alone presents no particular corrosion problem to aluminum alloys. However, there are certain circumstances under which corrosion of a metal can occur in the presence of moisture.

As shown in Figures 1 and 2, aluminum alloys have been found to exhibit a very high resistance to corrosion by seacoast and industrial atmospheres. As a result, the corrosion of aluminum automotive trim by the action of corrosive atmospheres is not a particular problem. There are several other conditions, however, that could exist in applications of automotive trim and that could create corrosion problems with most metals. These are galvanic corrosion caused by contact with dissimilar metals and poultice action brought about by the presence of water absorptive materials on the surfaces of the metal. Past experience has shown that these corrosive situations should not affect the good performance of aluminum automotive trim because of the present methods of installation, service conditions and maintenance procedures.

With respect to galvanic corrosion, it appears that aluminum automotive trim would probably contact

painted steel, chromium plated steel and stainless steel under service conditions. Aluminum alloys have been found to resist galvanic corrosion by contact with these metals very well. The effects of poultice action also do not appear to be a particular problem for aluminum automotive trim. The design of trim members is such that any crevices that might be present are small and the maintenance procedures followed on automobiles today are sufficiently frequent and thorough that the opportunity for the formation of a poultice condition on the aluminum surfaces appears rather remote. In addition, anodically coated aluminum alloys have shown a high resistance to corrosion by poultice action.

Maintenance

In general, aluminum alloy automotive trim requires only the ordinary maintenance provided by the car owner for good performance. The performance and appearance of aluminum trim, however, will vary to some degree according to the amount of care provided by the owner. In other words, the trim that has received regular maintenance will provide somewhat better performance than other trim that is not maintained or maintained only infrequently. The same variations in performance as affected by maintenance pertain to all parts of an automobile.

Ordinary road films can usually be removed from aluminum trim by washing with clear water followed by a thorough drying. In cases where dirt films are more difficult to remove, a wash with mild soap and warm water followed by a rinse and thorough drying can be employed. In addition to these methods, there are many satisfactory wax base and non-wax base polish cleaners available for use by the car owner. In isolated cases involving the accumulation of long-time dirt deposits, it may be desirable to use a non-etching type chemical cleaner applied according to the manufacturer's directions. This procedure also should be followed by a thorough rinse and drying.

One precaution should be taken in connection with the maintenance of aluminum automotive trim. The use of uninhibited alkaline cleaning solutions on aluminum should be avoided. Although available, these drastic cleaners are not only unnecessary but their severe cleaning action actually damages the finish on aluminum trim. The use of such cleaners also can have deleterious effects on the paint finish and other decorative members employed on automobiles. For that reason their use obviously is not desirable. Inhibited alkaline cleaners are available for use on aluminum alloys.

Service Experience

In addition to the accumulation of laboratory test data, service experience obtained over the last 15 years has indicated that aluminum alloys are well-suited to automotive trim applications. Probably the first commercial application of anodically coated aluminum for automotive trim was in buses. The first buses employing such aluminum trim were produced in 1940 using 5052 alloy sheet with anodic treatments of 18 to 23 minutes duration. Some of the original buses are still in operation with the aluminum members providing very satisfactory performance. Large quantities of anodically coated aluminum are still being employed in this application.

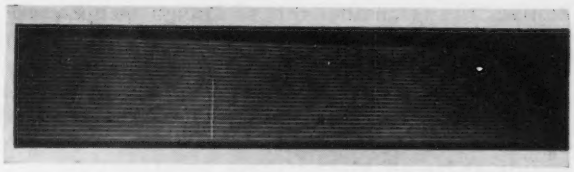

Figure 4—Photograph showing an anodically coated aluminum trim member used on a 1957 production model car.

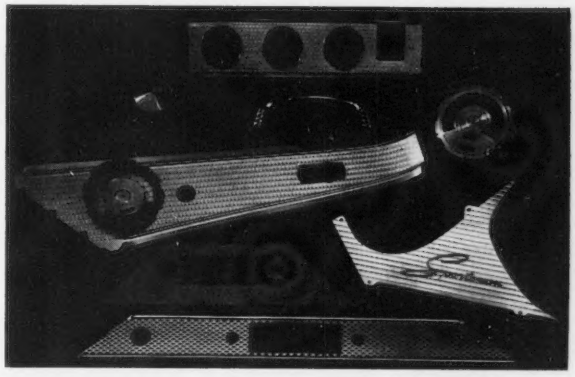

Figure 5—Photograph showing production aluminum interior parts for passenger cars. Included are chromium plated aluminum permanent mold cast grab handle, textured aluminum instrument panels, and anodically coated quarter panel trim and medallions.

Another early service application of aluminum alloys in the automotive field involved a 1942 production model car. This production model was altered by Alcoa so as to substitute aluminum alloy parts for many of the members commonly made of steel. These included window moldings, radiator grille louvers, windshield moldings, fender moldings, wheels, bumpers, trunk lid, door panels, hood and other miscellaneous parts. Various aluminum alloys, including some of those shown in Figures 1 and 2, were employed. This car was driven in the vicinity of Cleveland, Ohio until September, 1955, at which time the car (Figure 3) was dismantled and the aluminum parts examined in detail.

In general, the aluminum alloy members have performed very satisfactorily and were superior to the other conventional materials employed in the car. The aluminum body sections that had been painted to match the car were in excellent condition and vastly superior to any steel body section. The plain buffed finish of the aluminum alloy trim members lacked the original luster of the chromium plated parts. However, the finish of the aluminum parts was brighter at the end of the test than that of the chromium plated steel parts. From the standpoint of resistance to corrosion, all of the aluminum alloy parts, including the uncoated trim members, were superior to the chromium plated steel or the zinc alloy members.

Also directly associated with the subject of auto-

motive trim is another car, of foreign manufacture, using uncoated aluminum trim and hardware. Several of these parts were anodized for test purposes. Included in the anodized parts were outside door handles, outside escutcheon, inside door handles and door pull, window winder, front bumper, bumper guards and scuff plates. These members have now been in place on this car for approximately 3½ years and have maintained a very attractive appearance with very little, if any, corrosion.

In recent years, aluminum trim has become a standard item on several production model cars. Some cars are employing aluminum moldings and medallions. Others have employed license plate holders, grilles, wheels, bumper members, and, as was pointed out previously, window moldings. In all cases, very satisfactory performance from the standpoint of appearance and resistance to corrosion has been obtained.

The use of aluminum automotive trim is increasing on the 1957 model cars. Figure 4 illustrates one of these new uses. In addition to the exterior trim members, anodically coated aluminum alloys are being used extensively for interior trim applications. Included among these, as shown in Figure 5, are instrument panels that may be uncoated, anodically coated, or lacquered, and scuff plates which, incidentally, are being employed on all makes of automobiles at this time. One manufacturer also has employed chromium plated aluminum grab handles in his production models.

In addition to the service applications described above, many other small items made of aluminum are being used. From this summary, it is apparent that aluminum alloys are not foreign to the automotive trim field and that there are service data as well as laboratory test data available to indicate that aluminum alloys can be employed in this application with confidence.

Conclusions

Laboratory test data and field service have indicated that aluminum alloys are suitable for use in automotive trim applications. These alloys possess a high resistance to corrosion and are available in a wide variety of finishes suitable for the many different types of service required by the automotive industry. The high inherent resistance to corrosion of aluminum, together with the relatively simple protective measures that can be applied where necessary, make this metal appear to be highly desirable for diversified applications in automotive trim.

Acknowledgment

Appreciation is expressed to R. H. Brown, C. J. Walton and F. Keller for their helpful suggestions and contributions to this paper.

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Any discussions of this article not published above will appear in the December, 1957 issue.

TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

Corrosion and Metal Transport In Fused Sodium Hydroxide*

Part 1 – Experimental Procedures

By G. PEDRO SMITH, M. E. STEIDLITZ and E. E. HOFFMAN

Introduction

THIS PAPER, the first of a series, describes the corrosion testing techniques that were used in studies of corrosion and mass transfer of metals in fused sodium hydroxide at temperatures up to 815 C. The data that were obtained will be presented in subsequent papers. Previous research in this field was reviewed by Smith.[1]

Some of the factors which previous research has shown to be of importance in the design of fused hydroxide corrosion tests are the following:

1. Only one metal or alloy should be exposed to a hydroxide melt. When two different metals are exposed to the same fused hydroxide melt, it is frequently observed that one metal contaminates the other or that the two metals contaminate each other.[2] This process of metal transport, usually referred to as "isothermal mass transfer," is frequently observed in high-temperature corrosion and may be caused by a variety of liquid media.[3]

2. In conducting corrosion tests the hydroxide melt should be kept as isothermal as possible. A great many metals undergo mass transport in fused sodium hydroxide under the influence of a temperature differential.[1] This mass transport produces a spurious corrosion at the immersed metal surfaces which are hottest and may oppose corrosion at the metal surfaces which are coolest. In some cases a temperature gradient of only a few degrees is sufficient to produce a measurable effect.

3. In both corrosion and mass-transport studies with fused sodium hydroxide, control of the blanketing atmosphere is of critical importance. Metals react with fused hydroxides to give hydrogen, and it is frequently found that corrosion is slower in the presence of this blanketing gas.[1] Lad and Simon[4] have quantitatively demonstrated the inhibiting effect of hydrogen on the mass transfer of nickel. On the other hand, oxygen is an especially effective oxidizing agent in the presence of fused

* Submitted for publication April 29, 1957.

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E. E. HOFFMAN attended the University of Notre Dame where he received a Bachelor of Science degree in Metallurgy in 1951. Since graduation he has been associated with the Metallurgy Division of the Oak Ridge National Laboratory. His professional experience has been concerned primarily with corrosion problems encountered in the application of liquid metals as coolants for nuclear reactors operating at elevated temperatures. Mr. Hoffman presently heads the General Corrosion Group of the Metallurgy Division.

Abstract

Experimental techniques are described for studying corrosive reactions and metal transport in fused sodium hydroxide at temperatures up to 815 C. The apparatus described is designed for small-scale testing.

Some of the factors which previous research has shown to be of importance in the design of fused hydroxide corrosion tests are reviewed. Test methods discussed include the capsule test technique, cold finger test technique, and controlled-atmosphere test technique. 4.7

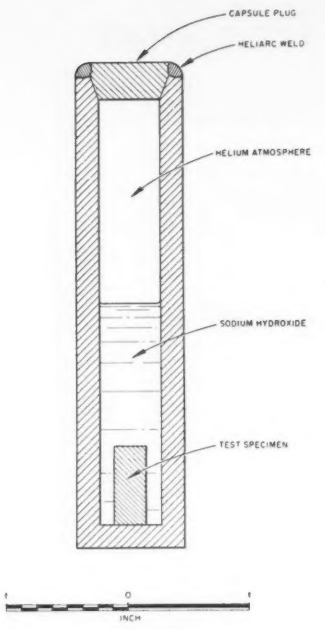

Figure 1—Cross-sectional diagram of capsule used for corrosion tests with fused sodium hydroxide. Both capsule and specimen were machined from the same bar of metal.

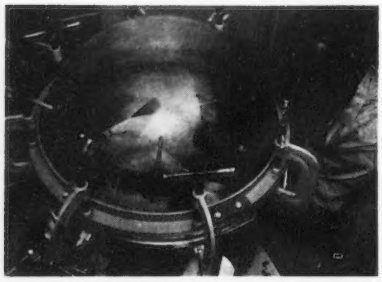

Figure 2—Purified helium-atmosphere chamber used for loading and welding capsules for sodium hydroxide corrosion tests. The technician is holding an electrode used for welding in the chamber.

sodium hydroxide and its presence will ensure accelerated corrosion.

4. Exposure of sodium hydroxide to air prior to use in corrosion tests should be minimized. Sodium hydroxide reacts fairly rapidly with atmospheric carbon dioxide to form sodium carbonate. Although the possible effects of carbonate contamination on corrosion and metal transport are largely unknown, Lad and Simon[4] have reported that sodium carbonate above 1 weight percent accelerated the rate of transport of nickel in fused sodium hydroxide.

Capsule Test Technique

The capsule test technique, which was used to obtain an initial evaluation of the corrosion resistance of a metal in a sodium hydroxide melt, consisted in heating a sample of the metal under test together with the hydroxide in a small sealed capsule also made of the metal under test. This technique was briefly discussed by Vreeland, Hoffman, and Manly[5] as a preferred procedure for "static" testing.

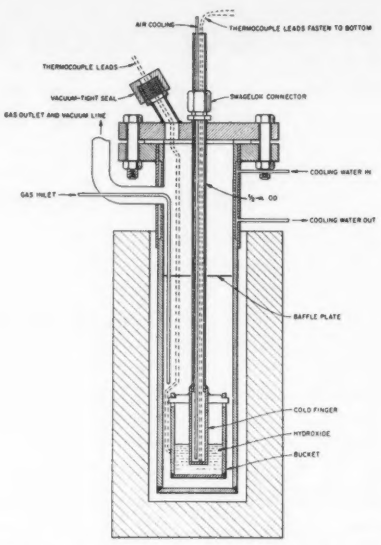

Figure 3—Cross-sectional diagram of the cold finger apparatus used to study mass transfer of metals through NaOH in the presence of a temperature gradient.

The capsule design and the capsule closure technique used depended on whether the metal under test was available in the form of tubing or of solid rod. When the metal was available in the form of tubing, the capsule test procedure of Vreeland, Hoffman, and Manly[5] was used. When the metals were available only in the form of solid rod, as was true in the majority of cases, capsule plugs and test specimens were machined as shown in Figure 1. The capsules were cleaned and then annealed in a hydrogen-atmosphere furnace, and the test specimens were electropolished and weighed. The capsules were then loaded with test specimens and sodium hydroxide, and the plugs were welded in place in a purified helium-atmosphere chamber. The sealed capsules were then enclosed in protective jackets as described for the tests with capsules made from tubing.[5]

Figure 2 shows the helium-atmosphere chamber used for assembly of the capsules. The plastic dome which covered the chamber allowed good lighting and visibility while withstanding the one atmosphere pressure differential produced when the chamber was evacuated during purging. A seal between the plastic dome and the chamber was accomplished by means of a 0.75-inch neoprene O-ring. The dome was hinged so that it could be opened for cleaning the chamber or for introducing large pieces of apparatus. The technician shown in Figure 2 is sealing a test capsule by welding. Manual operations in the chamber were performed through heavy-walled neoprene gloves, the cuffs of which were tightly sealed to the chamber with O-rings.

The hydroxide which was used in the capsule tests

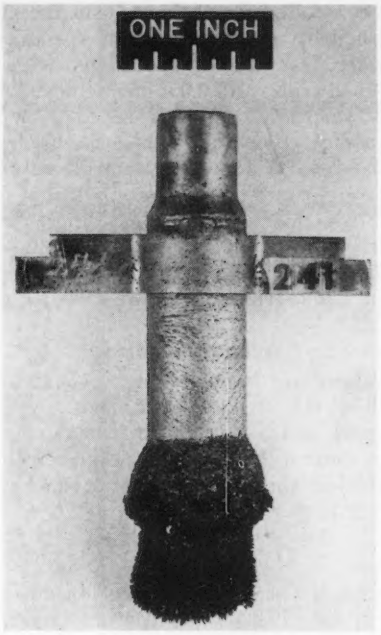

Figure 4—Cold finger made of Armco iron and having an encrustation of iron crystals on its lower end where it dipped into the hydroxide melt.

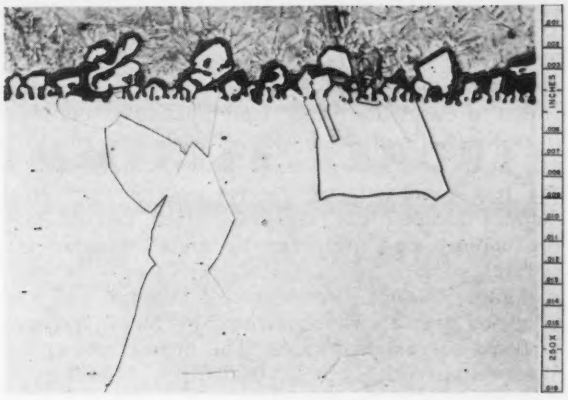

Figure 5—Cross section through a deposit of nickel crystals on a nickel cold finger. This deposit had a maximum thickness of 0.003 inch and was formed by mass transfer through fused sodium hydroxide.

was dehydrated according to the recommendations of Smith,[6] cast into sticks and stored in gastight jars until used. When the capsules were loaded, the hydroxide was added in a molten state. The capsules were filled with sufficient hydroxide to cover the 0.625-inch-long specimens to a depth of 1-inch. This loading gave a surface-to-volume ratio of about 4.5. All of the above operations were carried out in a helium-atmosphere chamber.

At the completion of a corrosion test the hydroxide was separated from the metal parts by being dissolved in water. In some experiments the aqueous hydroxide solution was analyzed for heavy-metal ions. Then various measurements were made on the metal specimens. In most tests, measurements were made of specimen weight change, specimen microstructure at and near corroded surfaces, and specimen thickness change. In some tests chemical and x-ray diffraction analyses were made of layers of corrosion product and metal from the surfaces of the specimens. Details of the utilization of these data for the evaluation of corrosion will be discussed when the results of the tests are reported.

Cold-Finger Test Technique

The apparatus which was used to study mass transfer under the influence of a thermal gradient is diagrammed in Figure 3. The sodium hydroxide was contained in a bucket, 2-inches in outside diameter, 3-inches in height, with a 0.065-inch-thick wall, and fabricated from the metal under test. The outside wall temperature of this bucket was measured by a thermocouple spot-welded in place. Arranged axially within the bucket was a tube, 0.75-inch in outside diameter and closed off at the bottom. This tube, referred to as a "cold finger," was cooled by a stream of air introduced through a small metal tube as shown in the diagram. The inside surface temperature of the cold finger was measured by a thermocouple spot-welded to the bottom. Both bucket and cold finger were fabricated from the metal under test.

Approximately 90 grams of sodium hydroxide was used in each test. This amount of hydroxide, when molten, immersed the cold finger to a depth of about 0.75-inch.

The temperature and temperature differences which will be reported were those measured by the thermocouples welded to the inside of the cold finger and to the outside of the bucket.

The bucket and cold finger were contained within a vacuum-tight chamber shown in Figure 3. The atmosphere which filled this chamber was introduced through a tube which reached a point near the top of the hydroxide. A slowly flowing stream of gas was maintained throughout each test.

The atmospheres used in the tests were helium and hydrogen. The helium was purified by passage through two columns packed with calcium turnings, the first at 700 C and the second at 400 C. The hydrogen was purified by passage over copper turnings at 700 C and over a calcium-calcium hydride mixture at 400 C.

The test procedure with the cold-finger apparatus was as follows: After fabrication, the bucket and cold finger were annealed in a hydrogen-atmosphere furnace. Then they were assembled in the test apparatus, and 90 grams of chemically pure grade sodium hydroxide was added to the bucket. The lid was bolted onto the test chamber, and vacuum pumping was started immediately. The hydroxide was dehydrated by heating to 400 C under vacuum for about 16 hours. Then the desired atmosphere, hydrogen or helium, was introduced, the temperature of the bucket was raised to the desired level, and, finally, cooling air was turned on inside the cold finger and adjusted to maintain the desired temperature differential between cold finger and bucket.

Figures 4 and 5 illustrate the kinds of results

which were obtained with the cold-finger apparatus. Figure 4 shows an Armco iron cold finger after test. The lower end of the cold finger, which was immersed in the melt, is encrusted with crystals of iron which grew while iron simultaneously dissolved from the walls of the bucket. The deposit shown in this figure was exceptionally heavy. Under most of the test conditions of interest only a very small amount of metal was mass-transferred. In such cases the deposit was measured by metallographic procedures.

Figure 5 shows a cross section through a deposit of nickel crystals which formed by metal transport in fused sodium hydroxide. The deposit shown has a maximum thickness of 0.003-inch. However, no difficulties were encountered in measuring deposits down to 0.0002-inch thick. In preparing metallographic sections it was necessary to protect the deposits with an electroplate before mounting in plastic in order to reduce rounding-off of the edge of the specimen during polishing. The details of measuring mass transfer by metallographic methods will be discussed in subsequent papers.

Controlled-Atmosphere Test Technique

By means of minor modifications it was possible to use the cold-finger apparatus to study the effect of atmosphere on corrosion in the absence of mass transfer. This type of test, referred to a "controlled-atmosphere test," gave the same kind of corrosion information that was obtained in capsule tests except that the controlled-atmosphere test allowed studies to be made of the effect of a hydrogen atmosphere on the corrosion rate.

In the controlled-atmosphere tests the cold finger was replaced by a group of eight specimens of the metal under test. These specimens were 3-inches long, 0.5-inch wide, and 0.035-inch thick. Each specimen was suspended from a separate wire which passed through a vacuum-tight seal. Each specimen could be separately removed from the fused hydroxide and brought into a cool region without disturbing the other specimens. Thus, in a single run it was possible to corrode specimens for various times at a single temperature. The bucket and all the specimens in any one run were made of the same aloy.

Acknowledgment

The authors are indebted to W. D. Manly, J. V. Cathcart, W. H. Bridges, and D. C. Vreeland for wise counsel and assistance in the development of the experimental procedures described here. The photographs in this paper were obtained in collaboration with R. J. Gray.

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Any discussions of this article not published above will appear in the December, 1957 issue.

Publication 57-21

NACE TECHNICAL COMMITTEE REPORT

Section Two

Bibliographies of Corrosion Products

By NACE Technical Unit Committee T-3B on Corrosion Products★

Abstract

Selected abstracts are given on the identification and composition of corrosion products on aluminum, lead and lead alloys, silver, tin and tin alloys, and magnesium. Included are 37 abstracts on aluminum; 19 on lead and lead alloys; 15 on silver; 26 on tin and tin alloys; and 21 on magnesium. 3.4.3

Introduction

BIBLIOGRAPHIES in this section were prepared by Technical Unit Committee T-3B on Corrosion Products. This work, the second in a series of technical committee reports,[1] is an introductory reference to the subject of "corrosion products" and is not intended to be exhaustively complete. Selected key references and some others of particular interest are given, omitting many which probably might well have been included. Some of those omitted will be added to proposed future bibliographies.

All members of Committee T-3B contributed. E. L. Simons assisted in coordinating work of the contributors and M. C. Bloom, E. L. Simons, H. P. Godard, N. A. Nielsen and R. T. Foley edited the material.

★ A. H. Roebuck, Continental Oil Co., Ponca City, Okla., chairman.

(1) Section One of "Bibliographies of Corrosion Products" appeared in *Corrosion*, **13**, 111t (1957) Feb.

Part 6—ALUMINUM*

Corrosion Products

Use of Boehmite Films for the Corrosion Protection of Aluminum. D. ALTENPOHL. *Aluminum*, **31**, 10, 62 (1955). The formation of boehmite oxide films on aluminum in boiling water and their use for corrosion prevention of black water staining are described.

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* Prepared by W. L. Fink, Aluminum Company of America; J. K. McBride, Aluminum Company of America; Hugh P. Godard, Aluminium Laboratories, Limited.

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Electronographic Study of Hydroxide Films on Metals. N. A. SHISHAKOV. *Zhur. Fig. Khim.,* **26,** 106 (1952). The most frequent hydroxide film on Al consists of boehmite with a = 3.70, b = 21.09, c = 2.86 Å, but the solution of Al $(OH)_3$ formed from Al and aq. $HgCl_2$ deposits a hexagonal AlOOH of the brucite type with a = 3.07 and c = 4.70Å; an apparently identical lattice was described by Steinheil and attributed to a cubic oxide Al_2O_3. (*Chem. Abstr.,* **47,** 6207 (1953)).

Stained Aluminium Kettles. H. H. SYMONDS. *Metal Ind.,* **80** (1), 5-6 (1952). On the black staining of aluminum tea kettles. (*Met. Abstr.,* **19,** 844 (1952)).

Filamentary Growths on Metal Surfaces—Whiskers. K. G. COMPTON, A. MENDIZZA AND S. M. ARNOLD. *Corrosion,* **7,** 327-334 (1951). Mention made of filamentary growth on an aluminum alloy. (*Metals Rev.,* **8,** 554 (1951)).

Concerning Structure of the Oxide Formed on an Aluminum Surface. M. S. BELETSKII. *Dokladz Akademic Nauk U.S.S.R.,* **75,** 551 (1950). Boehmite does not form during oxidation of aluminum but is a secondary reaction taking place between water and dispersed Al_2O_3 present on the surface. (*A.S.M. Rev.,* **8,** 280 (1951)).

Latvijas PSR Zinntnu Akad. L. LEPINA AND Z. OSE. *Vestis,* **35,** 35-46, (1950). Composition and structure of corrosion products on aluminum in potassium chloride solutions. (*Chem. Abstr.,* **48,** 517 (1954)).

Surface Problems of the Corrosion Behaviour of Metal. W. GUERTLER. *Metalloberflache* (A), **3** (7), 133-141 (1949). Corrosion depends on the nature of film of corrosion products on the surface. (*Met. Abstr.,* **18,** 116 (1950)).

Investigation of the Corrosion of Aluminum Alloys in Carbon Dioxide Pressure Vessels. P. SCHLAPFER, H. GAUMANN AND A. BUKOWIECHI. *Schweiz Arch. angew. Wiss. u. Tech.,* **15,** 316-324 (1949). The chemistry of the corrosion that takes place. (*Chem. Abstr.,* **44,** 5788 (1950)).

The Mechanism of the Formation of Films on Metal. U. R. EVANS. Pittsburgh Intern. Conf. on Surface Reactions. 1948 pp. 71-76. Film growth on metals, effect of temperature on film growth, cracks in films and their repair. (*Chem. Abstr.,* **42,** 8040 (1948)).

Mechanism of the Corrosion of Aluminum. J. M. BRYAN. *Chemistry and Industry,* 1948 pp. 135-6. Formation of film of oxide or hydroxide on the surface. Al reacts initially to produce a protective film. Al $(OH)_3$ relatively insoluble over a wide pH range reducing corrosion in weakly acetic and alkaline solutions. (*Chem. Abstr.,* **43,** 548 (1949)).

Examination of Conductor Cables of Almelec and Aluminum-Steel after 15 and 25 years in Service. J. HERENGUEL. *Rev. Aluminum,* **24,** 357-60 (1947). Black adherent deposit on Al cables contained 50 percent free carbon. (*Chem. Abstr.,* **42,** 2562 (1948)).

Electron Diffraction and Electron Microscope Study of Oxide Film Formed on Metals and Alloys at Moderate Temperatures: Stripped Oxide Films of Metals. R. T. PHELPS. *Ind. Eng. Chem.,* Anal. Ed., **18,** 391-400 (1946). Study of Films on Fe, Ni, Co, Cr, Mo, W, Cb, Al and Cu. (*Chem. Abstr.,* **40,** 4328 (1946)).

An Electron Diffraction Study of the Atmospheric Oxidation of Aluminum Magnesium and Aluminum-Magnesium Alloys. L. DE BROUCKERE. *J. Inst. Metals,* **71,** (3) 131-147 (1945). Discusses compositions of films formed. (*Met. Abstr.,* **12,** 398 (1945)).

The Action of Water on Aluminum. H. LICHTENBERG. *Korrosion u. Metallschutz,* **16,** 251-254 (1940). Traces of heavy metals such as iron and copper may accelerate corrosion in certain waters. Example of analyses of a brown film containing magnesium 29, ferric oxide 2 and copper oxide 0.1 percent in a badly corroded pipe containing water exceeding 82°. (*Met. Abstr.,* **9,** 314 (1942)).

Electron Diffraction Examination of Oxide Films on Light Metals (Mg, Al, Be). I. ITAKI, S. YAMAGUCHI. *Nature,* **144,** 1090-1091 (1939). Mention of oxide film of Al at room temperature is $\delta - Al_2O_3$ while at 500 C the only pattern found is that of $\alpha - Al_2O_3$. (*Met. Abstr.,* **7,** 115 (1940)).

Diminution of the Attack of Aqueous Sodium Sulfide Solution on Aluminum by Addition of Sulphur. H. LICHTENBERG, K. GEIER. Na_2S solutions used to desulphurize viscose fiber attack aluminum bobbins severely. The gray deposit on samples is mostly Al $(OH)_3$ but contaminated with sulfur. (*C.A.,* **33,** 2452[6] (1939)).

Action of Cold Aqueous Ammonia Solution on Aluminum Sheet of Normal and Super Purity. H. RÖHRIG, J. ROCH. *Aluminum,* **21,** 128-130 (1939) February. The high purity sheet corrodes more rapidly. The formation of Al $(OH)_3$ (the principal corrosion product) determines the severity of the attack. (*C.A.,* **33,** 3740[2]).

Electron Diffraction Examination of Oxide Films on Light Metals (Mg, Al, Be). I. ITAKI, S. YAMAGUCHI. The oxide film at room temperature is $\delta - Al_2O_3$ and at 500 C $\alpha - Al_2O_3$. (*Nature,* **144,** 1090-1091 (1939)).

Atmospheric Corrosion of Aluminum. G. SCHIKORR. Aluminum alloy containing Si 4.90 percent, Cu 3.90 percent, Zn 1.03 percent and Fe 0.70 percent showed severe corrosion after six months exposure in Hamburg harbor. The decomposition product contained SO_3 14.5 percent, Cl 9.6 percent and 19.0 percent moisture. It was concluded that the product was caused more by the high sulfur content from combustible gases than the salt spray. (*J. Inst. of Metals* (London), **5,** 593 (1938) November.)

The Blackening of Aluminum by Hot Tap Water. J. FISCHER, W. GELLER. *Zeitschrift Fur. Metallkunde.,* **30,** 192-195 (1938). The blackening of aluminum by hot tap water is traced to the presence of a critical concentration of alkali and oxidizing material (eg, O_2) in the water. The cause of the black appearance is discussed.

Corrosion of Metals by Phenols. F. H. RHODES, P. A. RIEDEL, AND V. K. HENDRICKS. It appears that metallic aluminum reacts directly with the dry vapor of phenol or cresol to form aluminum phenolate or aluminum cresylate, and that on heating, these salts decompose to form diphenyl ether or dicresyl ether. (*Industrial and Engineering Chemistry,* **26,** (1934)).

The Effect of Tap Water Containing Cu on Aluminum. L. W. HAASE. *Metallwirtschaft,* **12,** 1933. It has often been found that the corrosion product from aluminum corroded locally by tap water contained Cu. A deposition of metallic Cu from the water could take place only if the water contained H_2SO_4, and such is not the case. Neutral and slightly alkaline water containing small amounts of Cu forms a scale on aluminum in which Cu is uniformly distributed. The Cu content of scale at corroded spots is higher than in other parts of the scale, so that it is reasonable to believe that the pitting was not due to Cu contained in the water. It is suggested that such pitting of aluminum is caused by particles of metallic Cu being lodged mechanically in the aluminum and the galvanic action taking place between the Al and Cu. (*C.A.,* **27,** 3019[8]).

Corrosion Exfoliation on Aluminum Sheet. LEOPOLD PESSEL. Aluminum sheet exposed to continuous salt spray exhibits the phenomenon of corrosion exfoliation. Minute crevices, formed from rolling, are made the anodic areas because of the exclusion of O_2. The formation of a large volume of Al $(OH)_3$ takes place in these crevices, causing the blister-like raising of the adjoining metal. (*C.A.,* **25,** 1787[2] (1931)).

Action of $Ca(OH)_2$ Solution on Aluminum. G. ASSARSON. *Z. Anorg. Chem.,* 1930. The action of calcium hydroxide solution (0.040-0.125 percent CaO) has been investigated at 0-90°. Analysis and crystallographic measurements of the resulting crystalline deposits show that the following calcium aluminates are formed under appropriate conditions: Al_2O_3, 4 CaO, 12 H_2O; Al_2O_3, 3 CaO, 12 H_2O; Al_2O_3, 3 CaO, 6 H_2O. (British C.A. A 1262 (1930)).

Gas Space Luminescence in a Heterogenous Reaction. L. TROMMER AND M. POLANYI. *Z. Physikal Chem.* The action of chlorine on aluminum in the presence of Cu gives rise to a luminescence in the space surrounding the metal. The band spectrum of the light emitted was identified with that of cuprous chloride. The action of chlorine on aluminum gives rise to an active substance, which in the presence of chlorine excites the luminescence of cuprous chloride. The active substance is presumably a subchloride of aluminum ($AlCl_2$). (British C. A. A 520 (1930)).

Reaction between Aluminum and Bromoform. S. GAMBARJAN. Aluminum turn-

ings, suspended in chloroform, are converted by bromine into a black, voluminous material insoluble in all media. The change probably occurs through production of acetylene and action of aluminum bromide. After being heated in a vacuum at 200 C, the product retains much aluminum. Treatment of the product for aluminum bromide leaves residues containing aluminum, whereas an almost ash-free residue with 38 percent oxygen is obtained by treatment with acetone. British C.A. A 269 (1928)).

The Corrosion of Certain Metals by Carbon Tetrachloride. F. H. RHODES AND J. T. CARTY. *J. Inst. of Metals* #1, **35,** 82. Aluminum exposed to vapor of carbon tetrachloride (at its boiling point) disintegrates into a dark gray powder consisting of aluminum chloride and hexachloroethane. (London.)

Corrosion of Some Cast Aluminum Alloys and a Method of Protection. A. C. ZIMMERMAN. *J. Ind. and Eng. Chem.,* **17,** 59. The gelatinous precipitate, which usually accompanies the corrosion of aluminum and aluminum alloy parts when in contact with water or aqueous solutions, has been found by analysis to be largely aluminum oxide.

The Corrosion of Certain Metals by CCl_4. F. H. RHODES AND J. T. CARTY. *Ind. Engr. Chem.,* **17,** 909-911. Corrosion product obtained from action of dry CCl_4 is $AlCl_3$ and hexachloroethane. *(C.A.,* 19, 3244.)

Part 7—LEAD AND LEAD ALLOYS*

Corrosion Products

The Action of Cracked Gasoline on Lead. L. G. GINDIN. *Compt. rend. acad. sci. U.R.S.S.,* **20,** 561-4 (1938) (in English). Auto-oxidation of Baku and Grozny cracked gasolines caused strong attack of lead. About 60 percent of the corrosion product is $PbCO_3$. *(C.A.,* **33,** 3740[8], (1939)).

The Corrosion of Pure Lead and Lead-Rich Alloys. O. HECKLER AND H. HANEMANN. *Z. Metallkunde,* **30,** 410-15 (1938). In hot sulfuric acid a marked distinction is found between electrochemical and chemical corrosion of lead. The former occurs when the lead is in contact with a conducting body having low acid solubility and high over-voltage. Under these conditions a protective layer of $PbSO_4$ in the form of Anglesite arises. In the absence of such cathode areas, chemical corrosion produces white, nonadherent $PbSO_4$. This occurs in the presence of bismuth. *(C.A., 33, 3742[4],* (1939)).

Formation of Protective Layers and Corrosion of Lead. G. SCHIKORR. *Korrosion und Metallschutz,* **16,** 181-7 (1940). The progress of corrosion in water depends upon whether $PbCO_3$ forms on the lead surface or at some distance away. The attack by organic acids generally increases with the solubility of the corresponding lead salt. *(C.A.,* 35, 3216[2], (1941)).

Formation of Lead Monoxide as a Cable Sheath Corrosion Product. E. F. WOLF AND C. F. BONILLA. *Trans Electrochem. Soc.,* **79,** 307 (1941). Authors studied the conditions of voltage and electrolyte composition under which red tetragonal PbO is formed at room temperature. Low salt concentration, voltage below 1 volt, and low solubility of the Pb salt of the corroding anion favor red PbO formation. *(C.A.,* **35,** 3947[3], (1941)).

Behavior of Lead in Hot Sulfuric Acid of Moderate Concentration. G. SCHIKORR AND I. E. SCHALLER. *Metallwirtschaft,* **20,** 1135-8 (1941), Chem. Zentr. 1942, I, 1807. Attack in hot 30-80 percent H_2SO_4 was studied. Three types of corrosion are described. A. Slight attack with formation of a dark coating; B. Rapid attack with vigorous H_2 evolution; C. Complete decomposition with evolution of SO_2. Type of attack depends on composition of lead, acid strength and temperature. *(C.A.,* **37,** 3390[2], (1943)).

Corrosion of Lead—Indium Diffusion Alloys. J. M. FREUND, H. B. LINFORD AND P. W. SCHUTZ. *Trans. Electrochem. Soc.,* **84,** (1943). No protective film is formed on Pb in lubricating oil at 170-180 C. PbS is one of the corrosion products. Indium (plated and diffused or alloyed) inhibits the attack on lead. *(C.A.,* **37,** 6632[4], (1943)).

The Identificaton by means of X-ray Diffraction Patterns of the Products of Corrosion of Lead by Water in the Presence of Air. M. TABOURY AND E. GRAY. *Compt. Rend.* **217,** 211-13, (1943). The powder diffraction diagram is identical with that obtained from a mixture of cerussite ($PbCO_3$) and hydrocerussite ($2PbCO_3 \cdot Pb(OH)_2$). *(C.A.,* **38,** 5787[3], (1944)).

The Corrosion of Metals by Methanol. ALAIN GUILLEMIN. *Ann. Chim.,* **19,** 145-201, (1944). The corrosion of Al, Cu, Sn, Pb, Zn, Cd, Sb, Bi, Cr, Fe, Ni, Co, by methyl alcohol was studied at room temperature. Lead corrosion is reduced by 20 percent water content. Lead is attacked much less by 1 percent aqueous formic acid than by 1 percent formic in methanol. The formation of Pb $(OME)_2$ is attributed to initial surface oxidation. In absence of air, lead is not attacked by methanol. *(C.A.,* **40,** 1437[9], (1946)).

Solubility of Lead Fluoride in Aluminum Fluoride Solutions. T. R. SCOTT. *J. Council Sci. Ind. Research,* **20,** 114-21 (1947). The severe corrosion of lead by AlF_3 solutions is attributed to solubility of PbF_2, due to depression of fluoride concentration by formation of complex alumino—fluoride ions. *(C. A.,* **41,** 6456[1], (1947)).

Rate of Corrosion of Lead by Hydrocarbon Solutions of Organic Acids. D. TURNBULL AND D. R. FREY. *J. Phys. and Colloid Chem.,* **51,** 681-704 (1947). The effect of acid structure and concentration was studied. For acid solutions of .01 M or less, at 70-80 C, the direct products of corrosion are basic Pb salts ranging in composition from $PbA_{1.5}(OH)_{0.5}$ to $PbA_{1.2}(OH)_{0.8}$ where A is the acid radical. These compositions are not greatly affected by the nature of the solvent, by temperature, or by molecular weight of acid, but become more basic as the acid concentration is lowered. *(C. A.,* **41,** 6516[h], (1947)).

Corrosion of Metals in Hydrocarbon Solutions of Iodine. L. G. GINDIN AND M. V. PAVLOVA. *Doklady Akad. Nauk S.S.S.R.,* **69,** 377-80 (1949). PbI_2 was the main corrosion product of lead in benzene solution (.219N Iodine). An isooctane solution corroded lead several times faster. *(C. A.,* **44,** 1878[g], (1950)).

The Corrosion of Lead by Xylene Solutions of Lauric Acid and P-Quinone. C. F. PRUTTON AND J. H. DAY. *J. Phys. & Colloid Chem.,* **53,** 1101-17 (1949). The probable mechanism involves reaction of 1 mole of quinone, Pb, and lauric acid to give a quinone-Pb complex. Further reaction of complex with 1 mole of lauric acid gives hydroquinone and lead laurate. *(C. A.,* **44,** 913[a], (1950)).

Reactions Accelerating the Corrosion of Lead Imbedded in Cement. M. DODERO. *Metaux et Corrosion,* **24,** 50-6, (1949). Weak A. C. or D. C. and thermo-electric currents may accelerate corrosion resulting in the formation of PbO. Stronger currents may form PbO_2. *(C. A.,* **44,** 1389[f], (1950)).

Corrosion of Metals by Hydrocarbon Solutions of Fatty Acids. L. G. GINDIN AND V. A. KAZAKOVA. *Doklady Akad, Nauk S. S. S. R.,* **80,** 389-92 (1951). The corrosion rates of magnesium, iron, and lead, and the corrosion products formed, vary with the acid, its concentration, and the solvent. *(C. A.,* **46,** 80[e], (1952)).

Film Formations on Metals in Hydrocarbons. A. GEMANT. *J. Electrochem. Soc.,* **99,** 279-84, (1952). A radio-isotope was used to follow the accumulation of corrosion products on lead, copper, and zinc in transformer oils. *(C. A.,* **46,** 11063[h], (1952)).

Corrosion of Metals by Non Electrolytes. VIII Corrosion of Metals by Hydrocarbon Solutions of Carboxylic Acids. L. G. GINDIN AND V. A. KAZAKOVA. *Zhur. Priklad. Khim,* **24,** 958-69 (1951). The products of corrosion of lead and of magnesium vary with the nature of the solvent and the acid and its concentration. *(C. A.,* **47,** 4826[e], (1953)).

Corrosion of Copper and Lead by Hydrocarbon Solutions of Iodine. L. G. GINDIN AND M. V. PAVLOVA. *Ibid.,* 1026-32. Lead in a .2096 N solution of I_2 in C_6H_6 is corroded to PbI_2, and in contrast to copper, much more strongly in an iso-C_8H_{18} solution. *(C. A.,* **47,** 4826[e], (1953)).

Effect of a Lead Dioxide Film Upon the Corrosion of a Lead Anode. G. Z. KUR'YAKOF AND I. A. KORCHMAREK. *Ahur. Priklad. Khim.,* **26,** 921-4 (1953). Electrodes without silver lost more than 10 times as much lead as those containing Ag. The process of corrosion consisted of:

(1) Film formation of quadrivalent lead, and salts formed in the electrolyte, which fell off as a slime;
(2) Passage of Pb^{++} into the electrolyte and to the cathode. The addition of elements forming insoluble compounds thickened the film, retarding corrosion.

(C. A., **48,** 5679[e] (1954)).

X-Ray Study of the Action of Oleic Acid on Lead. J. J. TRILLAT AND S. BARBEZAT. *J. Recherches Centre Natl. Recherche Sci.,* **16,** 18-20 (1951). Influence of temperature on the above, as well as

* Prepared by D. L. Hawke, National Lead Company.

other factors, was investigated. Includes X-ray diagrams. (*Corrosion,* **10,** 5a (1954)).

Part 8—SILVER*

Corrosion Products

The Reaction of Silver Alloys With Sulfur in Mineral Oil II. Examination of Reaction Films and Mechanism of Reaction. R. T. Foley, R. W. Hardt and H. O. Spauschus. *J. Electrochem. Soc.* **101,** 6 (1954). Reaction product films grown on silver alloys by reaction with sulfur in mineral oil were examined by spectroscopic, X-ray, and electron-diffraction techniques. Interpretation of the results leads to a mechanism of reaction in which the rate-determining step during the steady phase is diffusion through a strained interfacial zone of constant thickness, comprised of the sulfide of the alloying element dissolved in β-silver sulfide, altering the number of normal lattice defects. The sulfide of thallium, which, unlike all the other alloying elements, yields a sulfide with a cation/anion ratio similar to silver sulfide, is exceptional.

Dry Corrosion: The Sulfuration of Silver. Haldun N. Terem (Univ. Istanbul). *Rev. Fac. Sci. Univ. Istanbul,* **18A,** 81-100 (1953) (In French). A detailed description of experiments relative to the action of H_2S on pure silver (silver samples being either wires or plates). The experiments were undertaken to elucidate the effect of humidity, air, and temperature (between 20 and 800°) on the reaction rate. The products of corrosion are crystals of Argyrose which develop preferentially on edges and curvatures of the samples. At ordinary temperature and up to 300° thin films develop (under no condition does the interference color exceed blue of the second order).

The Rate of Corrosion of Silver in Ferric Perchlorate Solutions. Cecil V. King and Frances S. Lang. (New York Univ., N. Y.) *J. Electrochem. Soc.,* **99,** 295-300 (1952). Ag was found to dissolve more slowly in $Fe(ClO_4)_3$ or $Fe(NO_3)_3$ solutions than in $Fe_2(SO_4)_3$ solutions. A thorough investigation of the effect of Fe^{2+}, Fe^{3+}, Ag^+, and SO_4^{--} concentrations in perchlorate solutions, and of stirring speed, temperature, and polarization, was made. The difference in Ag solution rate was ascribed to a stronger adsorption of $AgClO_4$ than of $AgSO_4$ on Ag, and to the existence of a sulfatoferric complex ion which reacted more rapidly with the Ag than the normal hydrated Fe^{3+} ion.

Immunization of Metallic Surfaces Against Aggresive Agents. Jean Loiseleur. *Compt. Rend.,* **234,** 260-2 (1952). Protection against oxidation is due to spontaneous formation of neg. complex ions at the metallic surface. Consequently the generation of this type of oxide will assure the protection of metals which form nonprotective oxides. By immersing polished silver plates in a dilute solution of stannous sulfate, a mol. layer of SnO_2. Ag^{--} (purple of Cassius) is deposited on the surface, which thus retains its reflecting power in the presence of H_2S.

Silver Sulfide. M. H. Hebb. Electrical Conductivity of Silver Sulfide. *Journal of Chemical Physics,* **20,** No. 1, 185-190 (1952). Silver sulfide is an N-type semiconductor in both α and β forms. Conductivity decreases with increased sulfur pressure.

The Tarnishing of Silver in Aqueous Solutions and Properties of the Layers of Tarnish. Walther Jaenicke (Max-Planck-Inst. Phys. Chem., Gottingen, Ger.). *Z. Elektrochem.,* **55,** 186-93 (1951). The formation and properties of films which form on Ag in aq. halide and S solutions were investigated. The overvoltage of the corroding Ag electrode was measured against a non-corroding Ag electrode. The tarnishing velocity in Br and I solutions is initially limited only by the diffusion within the solution and is proportional to the halogen concentration. After the formation of compact films the velocity becomes independent of concentration and follows the parabolic law. With Ag_2S the tarnishing velocity is determined by one of the reactions at the phase boundaries. It is proportional to the S concentration, although largely independent of the film thickness.

Corrosion of Silver by Potassium Cyanide Solutions and Oxygen. Vibeke Lund (Univ. Copenhagen). *Acta. Chem. Scand.,* **5,** 555-67 (1951) (In English). In the initial stage of the solution, H_2O_2 is formed as an intermediate. When the CN concentration becomes sufficiently low the H_2O_2 undergoes catalytic decomposition on the Ag surface. Part of the H_2O_2 is also consumed in further solution of the Ag. The Ag dissolves in an amount equivalent to the CN present and forms the $Ag(CN)_2^-$ complex. The rate of O consumption is independent of initial CN^- concentration within the range 0.05 to 0.2M. It is first order with respect to the O pressure. The activation energy is 3.2 kcal from 15° to 25°. The velocity is determined by the rate of diffusion of O in the solution.

Corrosion of Metals in Mixtures of Air with Hydrogen Sulfide and in Solution of Hydrogen Sulfide. I. S. Shklovskii. Trudy Inst. Fiz. Khim, Akad. Nauk. S.S.S.R. 2, *Issledovaniya po Korrozii Metal,* No. 1, 257-75 (1951). Tests were conducted at room temperature for up to 60 days. Ag, Sn, and 18-8 steel changed in appearance considerably, but the corrosion film was protective and the corrosion was only 0.2-0.6 mg/sq cm in 60 days. In water saturated with H_2S, and kept in contact with air, Cu, Ag, Zn, and Ni suffered little corrosion but changed in appearance.

Silver Alloys with Increased Resistance to Chlorine. J. Schatz. *Werkstoffe u. Korrosion* 1950, 1 (6/7), 248-249. The rate of formation of silver chloride films on fine silver exposed to gaseous chlorine increases parabolically with time and exponentially with temperatures and is dependent on the velocity of migration of Ag^+ ions through the adherent surface film. Scale formation eventually becomes so rapid that the pure metal is valueless for use in chemical equipment operating above 200-220 C. However, the outward migration of Ag^+ ions, and consequently the rate of formation of silver chloride, can be lessened by the introduction of foreign ions, of higher valency than silver in the silver chloride lattice. This can be done by additions of cadmium, lead, or zinc to the fine silver, but such additions must only be sufficient to produce solid solutions, and not two phase systems. Additions of 3-6 percent cadmium are most effective; such alloys do not form excessive amounts of silver chloride film even after 16 hours at 300 C and as their mechanical properties are approximately the same as those of pure silver, no difficulty is encountered in working or on using them as cladding materials.

Rapid Oxidation of Metals and Alloys in the Presence of Molybdenum Trioxide. J. L. Meijering and G. W. Rathenau. *Nature,* **165,** 240-1 (1950); cf. W. C Leslie and M. G. Fontana, *C. A.,* **42,** 8760d. (Phillips' Research Labs., Gloeilampenfabrieken, Eindhoven, Holland). The temperature dependence of the increase in the rate of oxidation of Cu, Cu-Al alloys, Ag, and Al-Ag alloys caused by the presence of MoO_3 was studied. MoO_3 affects the rate of oxidation of Ag and Ag-Al alloys below 400° although the nearest eutectic is near 490°. Examination of the surface of a corroded 4/96 Al-Ag alloy showed alternate layers of oxide and Ag. The data are consistent with a mechanism involving liquid MoO_3 (melting point 790°) hampering the formation of a protective oxide layer.

Resistance of Silver to Hydrogen Fluoride. K. W. Frohlich and Martha Gretchman. *Chemie-Ing.-Tech.,* **21,** 68 (1949). Experimental corrosion data for 40 percent HF and 99.98 percent Ag metal at 18, 50, 75, 100, and 115° were 0.002, 0.007, 0.007, 0.011, and 0.047 mm per year, respectively. The corrosion product contained AgCl.

The Oxidation of Silver-Copper Alloys. Ernst Justus Kohlmeyer and Karin V. Sprenger. *Z. Anorg. Chem.,* **257,** 199-214 (1948). Ag-Cu alloys containing 0-30 percent Ag are completely oxidized by O at 1230°; those containing more than 30 percent Ag form an Ag-rich regulus beside the oxides, Ag_2O and Cu_2O. Similar results are obtained on heating mixtures of the oxides to 1230°. Molten Cu_2O dissolves up to 44 percent by weight of Ag_2O in air at atmospheric pressure; the melting point of Cu_2O is lowered about 170 degrees.

The Mechanism of Oxidation of the Simplest Gases by Oxygen on Thin Silver Layers. S. M. Fainshtein (Karpov Inst. Phys. Chem., Moscow). *J. Phys. Chem.* (U.S.S.R.), **21,** 37-50 (1947). Thin Ag layers produced by sublimation on glass adsorb, at —183° and 0.01 mm Hg, about 5 mol. layers of O_2 assuming the geometric and the real area to be identical. About 0.25-0.5 of this O_2 cannot be removed by evacuation between —183° and 300°. After evacuation at —183° less O_2 is adsorbed in a second and subsequent experiment Ag layers prepared at —183° and brought to 0° have no adsorption capacity. Oxygen which cannot be removed by evacuating reacts with H_2 or CO at 7-40°, the reaction being more rapid (less than 1 hour) with CO. One mol. of adsorbed O_2 reacts with 1.2-1.7 mol. H_2 or CO. Oxygen adsorbed at 183° loses the capacity of reacting with H_2 after heating to 200°. Perhaps AgO forms and does not dissoc. be-

* Prepared by H. A. Finch and R. T. Foley, General Engineering Laboratory, General Electric Company.

cause in it the O atoms are not close enough to combine to O_2 mols. H_2 reacts also with O atoms on Ag crystals. Neither H_2 nor CO is adsorbed by Ag layers at —183°. From the rate of the reaction the probability of oxidation of a mol. striking the Ag-O complex is calculated.

Crystallite Orientation in Coating Films. III. Orientation of Silver Iodine Upon Silver Bromide. GEO. MARIA SCHWAB. (Inst. Chem. Agr., Nicolaos Canellopoulos, Paraeus, Greece). *Trans. Faraday Soc.*, **43,** 715-23 (1947); cf. *Colloid-Z.*, **101,** 204 (1942). Layers formed on a solid surface by a chemical reaction of the surface with its environment, e.g. AgBr films formed by reaction of (a) Ag surfaces with Br or (b) AgCl surfaces with KBr solutions will be designated "coating films." AgI films formed by exchange coating of AgBr in I^- solutions are examined by X-rays to obtain their lattice orientation. As long as they do not undergo recrystallization or polymorphous change, they belong to the hexagonal modification and grow upon the lattice of the basic Br^- crystals such that the hexagonal basis (001) coincides with the octahedral plane (111) of the Br^-. Both planes have the same closest hexagonal arrangement of atom and contain ions of one sign only. Because of this, the phenomenon differs from the formation of the other Ag halides on each other or on Ag. In the coating plane the distances of nearest neighbors differ by as much as 13 percent. This is probably due to the lack of polarity in the plane and of the similarity of the ion distances Ag-Br and Ag-I. This, caused by the deformability of I^- makes the formation of mixed complexes possible in the transition layer having the coordination no. of one of the lattices.

Corrosion of Silver by Bromine. R. WEINER. *Arch. Metallkunde,* **1,** 281-4 (1947). When pure Ag is exposed to Br an adherent film of AgBr is formed.

Part 9—TIN AND TIN ALLOYS*

Corrosion Products

J. J. TRILLAT, L. TERTIAN AND M. T. PLATTARD. *Comptes Rendus,* **240,** 526, (1955). Tin fused under low atmospheric pressure showed no oxide pattern until 430 C when stannous oxide appeared. Stannic oxide appeared at 630 C. If the tin was just fused and then cooled slowly, stannous oxide appeared.

S. C. BRITTON AND R. DE VERE STACPOOLE. *Trans. Inst. Met. Finishing* (1955), Adv. Copy No. 6. Tin-cadmium alloy coatings form corrosion products in the atmosphere and in organic vapours substantially of cadmium compounds. In salt-spray, the corrosion product is modified by tin compounds.

Peculiarities Observed in the Corrosion of Tin by Acetic Acid Dissolved in Organic Liquids. R. DUBRISAY AND F. ARLET. *Bull. soc. chim. France,* p. 314 (1954) (*C. A.*, **48,** 8611e (1954). By chemical and X-ray analysis SnO_2 is found to form by acetic acid dissolved in alcohol. Acetic acid in benzene appears to form stannous acetate.

Continental Can Company, Inc., Research Information Letter of January 4, 1953. On soldered seams of empty cans in storage, a white deposit of basic lead carbonate forms if temperature is high and relative humidity exceeds 70 percent.

H. J. PLENDERLEITH AND R. M. ORGAN. *Studies in Conservation,* **1,** 63, 1953. Ancient tin coins from Malaya had crust of corrosion product in banded layers of grey and brown colors. Analysis showed mainly stannic oxide, about 7 percent stannous compound (probably oxide), some sulfate, and some traces of iron, silica, etc.

Corrosion Resistance of Tin and Tin Alloys. S. C. BRITTON. Tin Research Inst., p. 72, 1952. Tin-zinc coatings form zinc corrosion products in organic vapors analogous to those formed on zinc.

J. WELSH. *Chem. and Ind.*, p. 551, 1952. Tin-lead alloy coatings on copper in flue parts of gas-burning appliances always have copper in the corrosion product, apparently owing to diffusion through the coating.

Corrosion Resistance of Tin and Tin Alloys. S. C. BRITTON. Tin Research Institute, p. 28, 1952. Aqueous sulfurous acid produces film of sulfide and, at highest concentrations, loose black sulfide.

T. K. Ross. *J. Appl. Chem.*, **2,** 526, 1952. Various surface active agents produced white or cream crusts of variable tin content. Although there is some organic matter, this is considered to be largely absorbed material. The main constituent is believed to be a hydrolysis product of tin complexes.

R. K. HART. *Proc. Phys. Soc., B,* **65,** 955, 1952. Electron diffraction patterns showed amorphous film up to about 130 C. As temperature was raised above this, first stannous oxide and finally stannic oxide were obtained.

S. C. BRITTON AND R. M. ANGLES. *J. Electrodepositors Tech. Soc.*, **27,** 293, 1951. Tarnish films formed on tin-nickel alloy coatings in hot water were very resistant to acid unless cathodic treatment was given. Suggested that a combined oxide of tin and nickel is formed.

L. W. HAASE. *Metalloberflache,* **2,** 166, 1948. In tap water containing sodium percarbonate, tin forms a layer of stannic oxide and, locally, some hydroxides which, if the solution is sufficiently alkaline, are converted to loose precipitate of hydrated stannates.

J. C. HUDSON AND T. A. BANFIELD. *J. Iron and Steel Inst.*, **154,** 229P, 1946. Tin-lead coatings exposed in industrial atmospheres became covered with a white coating believed to be lead sulfate.

G. DERGE AND H. MARKUS. *Trans. Am. Inst. Min. Met. Eng.*, **143,** 198, 1941. Tin with additions of more than 0.1 percent zinc formed a layer of carbonate, probably of zinc, in sodium carbonate solutions.

Corrosion Products of Tin and Tin-Lead Alloys. J. A. SMYTHE. *J. Inst. Metals,* **66,** 355-60 (1940). Analysis of corrosion products of ancient tin and tin-lead alloys. Samples corroded in earth analyzed 25 percent to 40 percent SnO. On further oxidation this yields SnO_2. Suggests intermediate product. Corrosion product on sample in sea water found to be stannic oxide with small amount of stannous oxide.

W. T. COOKE. *Australian Chem. Inst. J. and Proc.*, **6,** 58, 1939. Liquid or gaseous sulfur dioxide at low temperatures produced a crystalline solid believed to be stannous hyposulfite.

T. P. HOAR. *Trans. Faraday Soc.*, **33,** 1152, 1937. Products of local corrosion in chloride solutions are likely to be oxides with perhaps oxy-chlorides. These appear first as an adherent film and later as loose product at isolated spots.

C. L. BAKER. *Ind. Eng. Chem.*, **27,** 1358, 1935. In corrosion of tin by sodium silicate solutions, it is suggested that a silica film is formed on the metal.

Black Spots on Tin and Tinned Ware. S. BRENNERT. T.R.I. Pub. D2, 1935. Products of local corrosion in chloride solutions consist principally of stannous oxide with about 20 percent stannic oxide.

R. D. JENKINS. *Proc. Phys. Soc.*, **47,** 109, 1935. Films formed on tin just above melting point were almost entirely stannous, but at higher temperatures, they were stannic.

Black Spots on Tin and Tinned Ware. S. BRENNERT. *Tech. Publ. Int. Tin Res. and Dev. Council.* Series D, No. 2 (1935). Used chemical analysis and X-ray. Black spots caused by neutral or almost neutral salt solutions found to be stannous oxide. Black spots on dairy equipment were concluded to be 76 percent SnO, 18 percent SnO_2, 1 percent Fe_2O_3.

A. STEINHEIL. *Ann. Physik,* **19,** 465, 1934. Tin foil heated in air gave stannic oxide electron diffraction pattern, but some stannous oxide was observed.

W. L. BRAGG AND J. A. DARBYSHIRE. *Trans. Faraday Soc.*, **28,** 522, 1932. Tin foil heated in gas flame gave stannic oxide electron diffraction pattern.

Corrosion of an Ancient Tin Specimen. C. O. BANNISTER. *J. Inst. Metals,* **35,** p. 71 (1926). Corrosion product from ancient scabbard of very pure tin analyzed to be 43 percent hydrated stannous oxide; 55 percent stannic oxide.

C. O. BANNISTER. *J. Inst. Metals,* **35,** 71, 1926. Corrosion product on ancient tin specimen, recovered from earth, was mixed stannous and stannic oxides with some sulphate.

H. S. RAWDON. *Trans. Am. Electrochem. Soc.*, **39,** 227, 1921. Tin-lead boiler plugs suffered conversion of tin to infusible oxide.

Part 10—MAGNESIUM*

Corrosion Products

The Behavior of Magnesium in Alkaline Solutions. E. I. LEVITINA. *Zhur.*

* Prepared by D. A. Vaughn, Battelle Memorial Institute; E. L. Koehler, Continental Can Company, Chicago, Illinois.

* Prepared by F. N. Alquist and J. L. Wasco, Dow Chemical Company, Midland, Michigan.

Obshchei Khim., **24**, 216-18 (1954); (*C. A.*, **48**, 10407 h). The formation of MgO hydrate was observed.

KAZAMASA ONO, ET AL. Japanese Patent 3374, July 17, 1953. *C. A.*, **48**, 7444a. An alloy of magnesium and some noble metal as Ag, Ni, Cu, is heated to form a MgO surface for a secondary electron emission surface.

Mechanism of the Solution of Magnesium in Acidic Salt Solutions. E. J. CASEY, R. E. BERGERON. *Can. J. Chem.*, **31**, 849-67 (1953); (*C. A.*, **48**, 1775e). The effects of ionic strength, acid activity, temperature and salt type on the structure of a surface film of MgO or $Mg(OH)_2$ which is present on the surface of magnesium in presence of acidic salt solutions.

The Surface of Anodically Polished Magnesium. K. HUBER AND H. FLUCKIGER. Comite intern. thermodynam. et cinet electrochim. Compt. rend. reunion 1951. 123-7 (Pub. 1952 in German); (*C. A.*, **48**, 1855d). MgO and $Mg(OH)_2$ were detected on the surface of magnesium anodically polished in a H_3PO_4 bath.

Corrosion of Metals in Dry and Moist Hydrogen Sulfide. I. S. SHKLOVSKII. Trudy Inst. Fiz. Khim., Akad. Nauk. S.S.S.R, 2, Issledovaniya po Karrozii Metal., No. 1, 241-56'(1951); (*C. A.*, **47**, 5859h). MgS, MgO, and $Mg(OH)_2$ are the corrosion products when Mg is exposed to moist H_2S.

Organic Complexing Agents in Textile Processing. JOHN SINGER AND F. C. BERSWORTH. *Am. Dyestuff Reptr.*, **41**, 1951 (*C. A.*, **46**, 8377f). Versenes are used to prevent the formation of calcium and Mg silicate scale in peroxide bleaching tanks.

Dyed Hydrated Oxide Coating on Mg and Mg-Base Alloys. H. K. DELONG. U.S.P. 2,482,728, Sept. 20, 1949; (*C. A.*, **44**, 530e). A magnesium or a Mg-base alloy first subjected to a treatment to form a hard, dense protective coating of hydrated oxide and then buffed to a high gloss can be dyed by immersing in a dyeing solution.

Chemical Reactions of Intermetallic Phases. I. Corrosion of the Intermetallic Compounds Ag_5Al_3 and Mg_2Pb. E. E. CHERKASHIN, ET AL. *Zhur. Obshchei Khim* (J. Gen. Chem.), **19**, 798-804 (1949); (*C. A.*, **43**, 8341g). The formation of $PbO\text{-}2MgO\text{-}3H_2O$ is explained.

Chemical Generation of Heavy Magnesium Fluoride Protective Coating on Magnesium Alloys. M. STAECHE. *Arch. Metallkunde*, **2**, 99-102 (1948); (*C. A.*, **43**, 2148d). A primary coat of $Mg(OH)_2$ is formed which is changed to Mg fluoride by boiling in fluoride solutions. Excellent corrosion resistance under atmospheric conditions and in O_2-rich salt water was experienced.

The Protection of Magnesium Against Corrosion by Chemical Means and by Paints. H. WEIS. *Mitt. Chem. Forsch. Inst. Ind. Osterr I*, 42-4 (1947) (Salzburg, Austria); (*C. A.*, **42**, 4895i). The effect of alloying elements in magnesium on the protectiveness of the subsequent oxide coating is discussed. Mg will form insoluble compounds with many anions as fluoride, oxalate, carbonate, phosphate. These deposits will cause a CH_2O resin base point to fall off if the paint contains any free CH_2O.

Reaction of Water Vapor and Hydrogen Peroxide Upon Light Metals at High Temperatures. O. KUBASCHEWSKI AND H. EBERT. *Metallforsching*, **2**, 232-5 (1947); (*C. A.*, **42**, 4507c). At 640° magnesium reacts with water vapor and with H_2O_2 at an accelerated rate. The H_2O_2 is completely reduced to give MgO and H.

Dissociation Pressures of Metal Oxides Formed on Various Solid Metals. BENJAMIN LUSTMAN. *Steel Processing*, **32**, 669, 676 (1946); (*C. A.*, **41**, 332h). Oxides of magnesium are included in this series at temperatures to the melting point and extrapolated to the boiling point.

Rates of High-Temperature Oxidation of Mg and Mg Alloys. T. E. LEONTIS AND F. N. RHINES. Am. Inst. Mining Met. Engrs., Inst. Metals Div., Tech. Pub. No. 2003, 28 pp (1946); (*C. A.*, **40**, 4645[8]). Magnesium forms a protective oxide scale at low temperatures, a nonprotective loose scale at higher temperatures, and may undergo combustion close to the melting point.

New Developments in the Study of Surface Chemistry. E. A. GULBRANSEN. *Metal Progress*, **49**, 553-9 (1946). Oxidation of Mg and other metals were studied under controlled conditions. Diffraction patterns show typical structures.

An Electron-Diffraction Study of the Atmospheric Oxidation of Al, Mg, and Al-Mg Alloys. L. DE BROUCKIRE. *J. Inst. Metals*, **71**, 131-47 (1945) (Paper 987); (*C. A.*, **39**, 2474[8]). The character of films formed on Al, Mg and Al-Mg alloys were studied. These films were formed by heating at room temperature and up to the melting point. MgO was usually present in the film.

Reaction Kinetics of Magnesium. A. VYSKOCIL. *Chem. Listy*, **38**, 17-21, 27-33 (1944); (*C. A.*, **44**, 6327e). In alkaline solutions and in presence of chlorides, magnesium forms a black powder on the surface, possibly a sub-oxide of magnesium.

The Investigation by Electron Diffraction of the Corrosion of Metals. SHIGETO YAMAGUCHI. *Bull. Chem. Soc. Japan*, **18**, 53-91 (1943) (In German); (*C. A.*, **42**, 1864f). Studies were made of the film structure and crystal size of corrosion products of magnesium by water, alkali, Cl^--containing water, and 10N HI.

Electron-diffraction study on the Corroded State of Metal. Corrosion of Magnesium by Water. S. YAMAGUTI. *J. Chem. Soc. Japan*, **61**, 857-60 (1940); (*C. A.*, **37**, 64[6]). MgO and $Mg(OH)_2$ were detected as the corrosion products.

A Method of Preparing Oxide Film on Magnesium and Its Alloys. E. OGAWA AND Y. MARUYAMA. *Bull. Inst. Phys. Chem. Research (Tokyo)*, **19**, 489-90 (1940); (*C. A.*, **34**, 5035[9]). Magnesium is made corrosion resistant by heating for one hour in water or in water vapor.

Composition and Crystal Structure of the Product of Slow Oxidation of Magnesium in a Damp Atmosphere. R. FAIVRE AND A. MICHEL. *Compt. Rend.*, **208**, 1008-10 (1939). The black substance formed on magnesium by slow oxidation in air is shown to be boucite with Mg atoms inserted in the lattice. (*C. A.*, **33**, 4102[4]).

Surface Oxidation of Metals in Air T. N. KRYLOVA. *Izvestiya Akad. Nauk S.S.S.R. Otdel. Tech. Nauk.*, **1938**, No. 10, 89-112. The oxidation processes of Mg and several other metals were studied by means of the polarization method of Drude. The metals were subjected to temperatures of 20° to 500° and higher. (*C. A.*, **34**, 4034[5]).

Publication 57-22

SYMPOSIUM ON CORROSION BY HIGH PURITY WATER

A Contribution to the Work of NACE Technical Committee T-3F On Corrosion By High Purity Water

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571t

NACE TECHNICAL COMMITTEE REPORT

Measurement of Corrosion Products in High Temperature, High Pressure Water Systems*

By A. A. SUGALSKI[1] and S. L. WILLIAMS[1]

Abstract

The concentration of corrosion products and other impurities which produce undesirable radionuclides must be maintained at extremely low values in the primary coolant systems of nuclear power plants to keep the resulting radioactive dose rate below certain maximum values. In the development of purification systems for maintence of these low concentrations it is necessary to measure the concentration of the various impurities and their solubility as they exist at the primary coolant temperature. This paper describes the development of a high efficiency filter used for this purpose and gives data obtained from both radioactive and non-radioactive water systems. 4.6.5

Introduction

THE OCCURRENCE of corrosion products in the high temperature water coolant of nuclear power plants can produce consequences which will directly affect the design and operation of these plants. Corrosion products can deposit upon the fuel, decreasing the heat transfer rate with a resultant increase in metal temperature and corrosion. Similarly, deposition of corrosion products can occur on the heat transfer surfaces of the steam generating equipment.

Corrosion products depositing on the surfaces of close moving components can affect the operation of these components. The activation of corrosion products and the buildup of radioactivity in deposits of corrosion products will significantly affect maintenance operations and present another problem. This activation occurs as a result of circulating corrosion products being exposed to nuclear fluxes as they pass through a reactor and also because of the release of radioactive corrosion products from the structural materials of the reactor itself. Due to the transport of radioactive corrosion products by the coolant, corrosion films located in non-nuclear portions of the steam generating system have built up sufficient activity to produce dosage rates exceeding 1 R/hr at the outer pipe surfaces.

The occurrence of corrosion products in the feed water cycle of conventional steam generating systems also can produce consequences which will affect the design or operation of these plants. Reference 1 reports an instance where the boiler feed water pumps lost up to 20 percent of their capacity due to the deposition on the pump internals of a sufficiently thick film of corrosion products to interfere with the flow.

Measurement of the concentration of corrosion products in the coolants of nuclear and non-nuclear power plants is necessary under different conditions of operation and with various structural materials in order to quantitatively evaluate the effects of these conditions on plant performance.

Conventional methods of sampling from high temperature water systems to obtain measures of corrosion product concentration have not proved particularly accurate. These methods which in general consist of drawing a 100 or 500 cc sample from a sample cooler and measuring conductivity, total solids or total iron may be sufficiently accurate where large quantities of corrosion products are involved, but in nuclear systems the total corrosion product concentration may be less than 1 ppm and still have a significant effect on plant operation. In addition the effect of temperature is important. In nuclear systems with water temperatures in the order of 500 F, the form of the corrosion products at operating temperatures may be greatly different from those at room temperature. Significant concentrations of colloidal iron or aluminum may be present in the coolant at operating temperatures producing extensive deposition on fuel or plugging of purification equipment and yet a cold sample may show only the presence of larger particles. It is therefore important to be able to measure small quantities of corrosion products circulating in the coolant accurately and to ascertain the form in which they are occurring at operating temperature.

This report describes the development of a high efficiency filter unit which was conceived in order to measure low concentrations of corrosion products circulating in nuclear systems and to separate the particulate corrosion products (1 micron and greater) from the colloidal

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(1) Knolls Atomic Power Laboratory, Schenectady, New York.

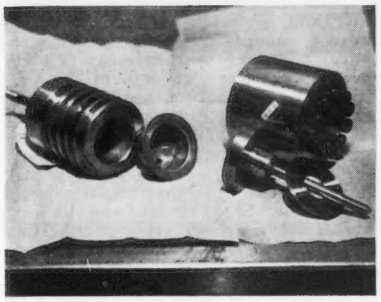

Figure 1—A high efficiency filter unit.

and soluble products at operating temperatures.

Several methods come to mind for obtaining a sample from a high temperature water system. First, one might use a bomb placed in a bypass line through which the hot water is circulated and then isolated to trap a sample. The objections to this procedure are that an unpredictable amount of adsorption takes place on the walls of the bomb during the flushing procedure. Precipitation or further adsorption may take place as the water in the bomb cools so that the water which is removed from the bomb is no longer representative. Neither would scrubbing or dissolving the precipitated material add to the representativeness since material present from the flushing operation undoubtedly would be present.

Removing the sample directly from the system is not feasible because it is necessary to cool the water for analysis. Cooling causes changes in solubles and colloidals concentrations and leads again to unrepresentative samples.

Cooling the water in a bypass line prior to passing it through a filter has the same disadvantage as cooling during sampling.

It became evident upon reviewing the various methods for withdrawing samples from high pressure systems that the water should be filtered hot. A sintered metal filter which immediately comes to mind for this purpose was found to be unsatisfactory because of the poor efficiency or plugging. To improve the efficiency and reduce the probability of plugging, a filter consisting of a sintered metal filter on which is mounted a pure graphite precoat was conceived. The testing of the precoated filter was carried out first on bench scale to determine its efficiency and performance at room temperature and 200 F and in a high temperature water loop to evaluate its performance at the more extreme operating conditions. The testing is described below.

Procedure (Bench)

A nickel sintered filter having a mean pore size of 5 microns was supported between teflon gaskets in a glass pipe flange so that the effluent could be collected in a vacuum filter flask. By maintaining a reduced pressure in the filter flask, it was possible to obtain a flow of ~0.05 gallons/minute. The filter was precoated with seven grams of graphite. After washing with one liter of water through the filter at the stated flow rate, the graphite was recovered from the filter, dried, and weighed. The gain in weight of the sintered metal filter was taken to be due to imbedded graphite.

The effluent was filtered through an analytical grade filter. The weight gain of this crucible was taken to be due to graphite not removed by the sintered metal filter.

A similar procedure was used for the insoluble corrosion product removal efficiency tests. Commercial Fe_3O_4 (which simulates the corrosion product of interest) having an average particle size of 100 percent 1-10 microns was used. One hundred mg of magnetite was slurried and filtered on a precoated filter. That magnetite which remained in the graphite was determined by ignition of the ashless precoat. Magnetite passing through the filter was determined by chemical analysis of the effluent. The loss was assumed to be imbedded in the filter. Chemical removal of this imbedded magnetite cannot be undertaken because of the possible attack of the sintered metal filter.

The results of the above tests are tabulated in Table 1.

The difference in filter efficiency for the various size graphite precoats is not significant. Also there is no significant difference in the result for the high temperature run.

From the amount of material found in the effluent and that found in the metallic filter, an efficiency of 99+ percent is indicated. However, the difficulties in quantitatively transferring material under the condition of the test prevent the obtaining of this high a recovery. Approximately 3-4 mg of material is actually not accounted for and therefore limits the efficiency value which can be stated with certainty.

The efficiency of this type of filter is better than 95 percent and most likely approaches 99+ percent.

The performance of the precoated filter is much more difficult to evaluate at the more extreme operating conditions of temperature and pressure. However, the results obtained with a single precoated filter were compared with those obtained using a precoated filter composed of two sintered disks between which were contained an acid washed paper filter. The results from the first unit compared favorably with those of the latter more efficient assembly.

Analytical Procedure

The procedure for determining waste weight is described below.

The entire filter and precoat is dried at 110 C for four hours. The graphite is carefully brushed into a tarred vial and weighed. The graphite is thoroughly mixed by rolling the vial on a Mini-mill for one-half hour. A two gram portion of graphite is then ignited for six hours at 1000 C in a platinum crucible. The total amount of residue is calculated for the filter

$$\frac{\text{grams ash} \times \text{grams total graphite}}{\text{Grams sample}} = \text{grams total insoluble corrosion products}$$

TABLE 1

Run No.	Test Material	Percent on Filter	Percent in Effluent	PERCENT IMBEDDED	
				By Weight Gain	By Difference
1	100 mesh graphite	99.4	0.01	0.29	0.59
2	100 mesh graphite	99.6	.07	.12	.33
3	100 mesh graphite	99.8	.00	.01	.02
4	100 mesh graphite	99.8	.01	.07	.19
5	100 mg magnetite on 100 mesh precoat	95.5	.02	.08	4.5
6	200 mesh graphite	99.7	.01	.07	.3
7	100 mg magnetite—200 mesh precoat	96.8	.004	.02	3.2
8	100 mg magnetite—no precoat	27.0	19.0	48.0	54.0
9	200 mesh graphite—200 F water	99.7	.01	....	.3
10	200 mesh graphite + 100 mg magnetite in 200 F water	97.0	0	.03	.30

TABLE 2

Time, Minutes	Rate, gpm	Filterables, ppm	Filtrate Fe, ppm
15	.05	6.5	4.2
15	.05	8.2	9.2
12	.05	3.8	1.4
15	.05	5.1	
15	.05	2.5	1.)
15	.05	4.1	.25
15	.05	2.6	.4
30	.05	4.0	.25

TABLE 3—Corrosion Product Concentrations and Filterables

Time Minutes	Rate, gpm	Filterables, ppm	Location
120	0.1	0.41	Entering pile
120	0.1	0.35	Leaving pile
120	0.1	0.033	Entering pile
120	0.1	0.035	Leaving pile

TABLE 4—Corrosion Product Concentrations —Non-Filterables in Parts Per Million

Fe	Mn	Cr	Co	Location
.091	.006	<.001	<.0016	Entering
.088	.013	<.001	<.0016	Leaving
.042	.002	<.001	<.0016	Entering
.077	.004	<.001	<.0053	Leaving
.044	.002	<.001	<.0016	Entering
.022	.002	<.001	<.0016	Leaving

A blank on the unused graphite of this purity is negligible.

The filter unit which evolved is shown in Figure 1. It consists of an autoclave type head closure in which is held a filter basket, fabricated so that the rim acts as a gasket. Since the corrosion products which were being measured did not include nickel, all parts of the filter unit exposed to the high temperature water were either fabricated from or plated with nickel. The spectrographic grade graphite was applied to the filter basket as a paste.

Test Apparatus

Recirculating Loop Description

Most of the work connected with the development of the hot corrosion product filter was carried out using a recirculating water loop operating at 2200 psi pressure and about 550 F temperature. The facility was constructed of one inch Schedule 80, A-106, Grade B carbon steel pipe. Immersion heaters were used to maintain the temperature and a steam pressurized to maintain the pressure. Ballast in the form of wire compacts also fabri-

cated from A-106, Grade B carbon steel were included to add approximately 500 square feet of corrodable surface area to the system. The flow through the main loop was about 18 gallons per minute.

A bypass loop was made of ½ inch carbon steel tubing and consisted of a cooler and mixed bed ion exchanger. The flow through the bypass circuit was 0.05 to 0.1 gallon per minute. The ion exchanger was used to purify the loop water periodically but was not operated during the hot waste filter tests.

Hot Corrosion Product Filter Installation Description

The filter was installed in the bypass circuit with a sampling line immediately below to allow the hot filtered water to be sampled. A gold or nickel lined vessel was attached directly to the sample line so that the 550 F water would not flash as it entered the sampling vessel.

Early experience indicated that care should be taken in positioning valves and fittings to reduce the deposition of particles in the low velocity regions caused by these components. In most later installations, valves were placed in vertical runs and velocities near those existing in the main loop piping were used.

Procedure (High Pressure Loop)

The graphite precoat was applied to the sintered metal filter as a paste to a depth of about ⅛ to ¼ inch. Flow was started through the filter at a rate of 0.05 gpm for a measured period of 15 minutes in the early tests to 60 or 150 hours during actual operation in in-pile facilities. Samples of water were taken downstream of the filter during filter operation.

Table 2 contains data taken at different times under different conditions of pH, ion concentrations, etc., on corrosion product concentrations obtained with the unit in the high temperature loop. It can be noted that significant quantities of iron passed through the filter unit, which demonstrates the value of the high temperature filtration for measuring the relative concentrations of particulate and non-filterable corrosion products.

Samples taken during a 30-day run under constant water conditions showed good reproducibility. All of the samples taken during this time remained between 1.1 and 4.0 ppm filterable solids concentration.

Several filter units have been installed in in-pile loops and data are slowly becoming available on the behavior and character of corrosion products in such facilities. Table 3 gives values of filterable concentrations measured up and downstream of an in-pile section of a carbon steel loop. Non-filterables concentrations measured in samples obtained from an in-pile loop using a hot filter are given in Table 4.

Discussion

This high efficiency filter has many applications in the study of corrosion products and their behavior, and is already being put into use by various groups working in the nuclear field.

In the development of control drive mechanisms, the effects of circulating corrosion products must be considered. These effects can be ascertained by maintaining various concentrations of corrosion products in the test facility by use of ballast steel or by introducing magnetite.

The concentrations are monitored and controlled through the use of the filter described. This type of filter provides a standard by which the efficiency of corrosion product removal by various purification media may be compared. Filters installed upstream and downstream of filter and ion exchange columns have given a quantitative measure of the removal efficiency of the test media for various corrosion products in the particulate, colloidal and soluble form. These filters have proved useful in investigating the applicability of various metals such as aluminum and titanium to high temperature water work, by providing a measure of the corrosion products released to the coolant and the form of these corrosion products at operating temperatures.

The filters have been used to study corrosion product deposition and release in reactor cores by installing the units upstream and downstream of the core and measuring both the concentration and specific activity of various corrosion product nuclides entering and leaving the core as particles and as colloids and solubles. These filter units also can be used for studying corrosion product deposition in the feed water cycle of conventional steam generating system, particularly where colloidal mechanisms are suspected or where the effect involves the local buildup of corrosion products over a period of time from very small amounts actually circulating in the system. The filters can be used also for studying both corrosion product behavior and coking in organic coolants.

In measuring concentrations of circulating coke particles in dry organic coolants such as bi-phenyl, high purity sodium chloride crystals or similar materials can be used instead of graphite.

Acknowledgment

The authors wish to acknowledge the efforts contributed by W. F. Mathewson, Jr. and E. L. Shirley to this work.

Reference

1. E. G. Gothberg, H. Kehmra and E. S. Johnson. Preliminary Investigation of Oxide Deposition in Boiler Feedwater Systems. ASME Paper No. S3-SA-19.

NACE TECHNICAL COMMITTEE REPORT

Corrosion of Aluminum-Nickel Type Alloys In High Temperature Aqueous Service*

By F. H. KRENZ*

Abstract

Aluminum can be made resistant to high temperature water by small alloying additions of Ni, Fe and Cu. In the presence of adequate concentrations of the cathodic second phase formed by these additions, the high temperature attack changes from grain boundary penetration and blistering to a more desirable uniform overall attack. The latter causes two films to be formed on the corroding surface, Film A growing outward from the original surface and Film B growing inward into the metal. Film A reaches a limiting thickness after 2-3 weeks at 300 C, but Film B continues to grow linearly with time. At this stage the corrosion rate is acceptably low ($\sim 1 \times 10^{-3}$ inches/yr) but the corrosion film is 10-20 μ thick, relatively brittle and subject to severe mechanical damage in water flowing at high linear velocities. Reactor radiation has an insignificant effect upon corrosion. Heat fluxes of the order of 100 watts/cm² also appear to have little effect, though in this instance the effect may have been obscured by mechanical damage.

Extensive test data are reported on the corrosion resistance in high temperature water of three specially selected aluminum alloys. 4.6.5

Introduction

DRALEY'S RECENT discovery[1] that alloys of aluminum containing small amounts of nickel and iron are resistant to corrosion by water at 250-350 C has aroused widespread interest in these alloys as possible materials for power reactor construction.

Ordinary 2S aluminum has surprisingly good corrosion resistance in water at temperatures up to 200 C. Above this temperature, however, the aluminum is rapidly penetrated intergranularly. Blisters formed in the metal adjacent to the surface disrupt the surface layers and break the protective film, thus greatly accelerating the attack.[1] Additions of nickel and iron to aluminum form a nickel-iron rich second phase cathodic to the aluminum matrix. The particles of this cathodic second phase, if properly distributed in the alloy,[2] have the property of changing the localized attack at temperatures above 200 C to a uniform penetration of the surface, producing an unbroken oxide film. Corrosion rates of such alloys are in the region of 10^{-3} inches/year in static water at temperatures as high as 350 C.

An application of particular importance involves the use of alloys of this type to sheath fuel elements in a water cooled reactor operating at temperatures in the neighborhood of 250 C. In a typical instance, the elements would be supported in horizontal or vertical cooling channels through which water would flow with linear velocities of 18-20 ft/sec. Sheath surface temperature would be approximately 300 C, and a typical heat flux through the sheath would be of the order of 100 watts/sq cm.** Such an application would impose on the alloy the most stringent demand for corrosion resistance. Other less demanding applications, such as fuel sheathing in water moderated reactors cooled by thermal convection, in reactors operating at lower temperatures, etc., could be enumerated.

At Chalk River, development of Al-Ni type alloys has taken the following lines:

(a) Tests have been made to determine the effect of alloy composition on the corrosion resistance of relatively complex ternary and quarternary aluminum alloys containing Ni, Cu, Fe and Si.

(b) Measurements have been made of long term corrosion rates of the more promising alloys in static and flowing water at 250 C-300 C.

(c) Tests have been made of prototype fuel elements clad in the most resistant Al-Ni alloys.

(d) An investigation into the nature of the high temperature corrosion process has been started.

The report which follows describes part of this work and discusses some of the fundamental aspects of the corrosion problem.

Experimental Techniques

Preparation of Samples

The alloys used in this work were cast as 10 pound tilt mold ingots, preheated to 495 C for 12 hours, scalped and hot-rolled to ¼ inch gauge, cold rolled to 1/16 inch gauge and sheared to size. They were annealed at 425 C for two hours followed by slow cooling

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* Atomic Energy of Canada, Limited, Chalk River, Ontario.

** 1 watt/cm² = 3172 BTU/ft²/hr.

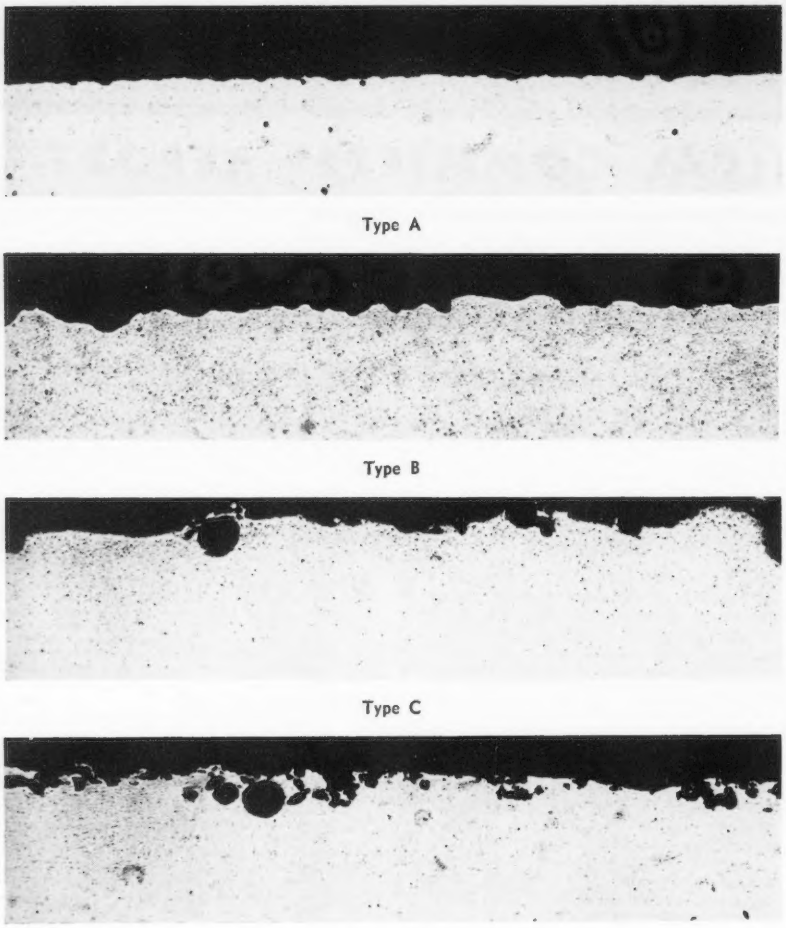


Figure 1—Polished cross sections of four corroded Al-Ni alloys illustrating the four categories of attack. Original magnification, 100X; actual magnification after reduction for engraving purposes, 85X.

(30 C/hr) to 260 C and thereafter air cooled.

Static corrosion tests were performed on samples 1 inch x 1.5 inch cut from the 1/16 inch sheet. Samples were cut to size, marked for identification and annealed for one hour at 450 C. Just prior to testing they were degreased in acetone, pickled, washed, dried and weighed.

Tests in flowing water were made on samples cut from sheet, tubing and bar stock. One-inch extruded bar was made from billets cast for this purpose. Tubing was made from the extruded bar by cold drawing on a mandril.

Methods of Testing

Static corrosion tests were made in standard one liter autoclaves of Type 316 stainless steel, half-filled at room temperature with distilled, deionized water. The samples were suspended from Type 316 stainless steel supports, no attempt being made to insulate them from the supports or autoclaves. No significant galvanic effects were found, and it was assumed that electrical insulation was unnecessary. The water was boiled briefly before sealing the autoclave in order to remove the bulk of the dissolved air.

The static tests were run at 300 C for intervals of one week, at the end of which time the samples were removed, examined and weighed, and the autoclave was filled with a fresh charge of water. The water quality generally varied from 1 megohm-cm specific resistance at the start of the test to 50-100 K ohm-cm at the end of the week. The pH usually started at 6.5-7.0 and finished at 7.5-8.0. This variation in test conditions was undesirable, but was accepted for tests merely intended to give semi-quantitative results and to compare performance of different alloys. More reliable results were obtained from loop tests in which the quality of the water was continuously controlled.

"Loop," in this report, refers to a closed circuit of high pressure tubing in which water is recirculated by a pump. Tests leading up to the design of the specimen fuel elements were performed mainly in the out-of-reactor test section of the "HTP" loop at Chalk River.[3] This is a typical stainless steel loop comprised of two parallel circuits supplied by a single pump, surge tank and heater. One circuit contains an autoclave, or test section, in an experimental hole in the Chalk River NRX reactor. The other circuit contains the out-of-reactor test section.

The test temperature in the HTP loop was 260 C (500 F). Tests were run

TABLE 1—Summary of 48 Hour Static Tests at 300 C on Al-Ni-Cu Alloys Based on Alcan 1S Aluminum

Weight Percent Ni	WEIGHT PERCENT Cu				
	0	0.5	1.0	2.0	4.0
0.5	2.85 D	(2.12) DC	1.00 C	1.18 BD	1.52 B
1.0	2.14 D	1.36 D	1.21 B	1.14 AB	1.16 A
2.0	1.21 BD	1.44 B	1.46 AB	1.02 A	1.22 A

Note: Numbers = mg/cm² wt. gain; Letters = type of attack.

at linear flow velocities of 18-20 ft/sec. The loop was pressurized to 1000 psi to prevent local boiling and cavitation. Test periods varied from two to six weeks.

The specimen fuel elements were tested in an X-loop in the NRX reactor.[4] This is a standard stainless test loop in which the test section occupies one of the fuel rod positions of the reactor. Test conditions in this loop were as follows: temperature 288 C (550 F), static pressure 2000 psi, linear flow velocity 18-20 fps.

Evaluation of Results

The test specimens were examined in several ways before making an assessment of corrosion resistance.

Visual examination. The appearance of the corroded specimen under the stereomicroscope (25-100 x) was usually a good index of corrosion resistance. A resistant alloy was characterized by a smooth, adherent corrosion film without pits, cracks or blisters.

Measurement of weight gain. Simple measurement of weight gain (mg/cm²) as a function of total time of exposure often gave a reliable index of corrosion rates. Weight gains could not, of course, be relied upon in cases where the film was obviously non-adherent.

Measurement of weight loss. These measurements were made on samples stripped of corrosion film, using a technique perfected by Draley and Ruther.[5]

Measurement of dimensional changes. Micrometer measurements of sample dimensions proved particularly useful in instances where corrosion rates were high, corrosion was non-uniform, or specimens were intensely radioactive.

Microscopic examination of polished cross-sections. Polished cross-sections of corroded specimens revealed the nature and extent of attack, the metallurgical condition of the alloy at the point of attack and the character and thickness of the corrosion film. This examination provided the most reliable information about the specimen, and at least one polished cross-section was examined before a final assessment of the corrosion resistance of an alloy was made.

For convenience in summarizing the results of screening tests made on a large number of alloys, the following types of attack were recognized:

Type A. Metal surface uniformly penetrated, covered by a uniform film free from cracks and imperfections.

Type B. Surface mildly pitted, film less uniform.

Type C. Pitting occasionally severe. Film uneven, flaky or cracked.

Type D. Severe attack, Typical "blisters" found underlying the surface. These are roughly spherical cavities which erupt at some stage of corrosion and cause breaks in the film. Blisters were first described by Draley[1] who has suggested that they are formed as a result of H-atom penetration into the metal.

Figure 1 shows cross sections of corroded samples classified according to the types of attack just defined.

Results

Effect of Alloy Composition

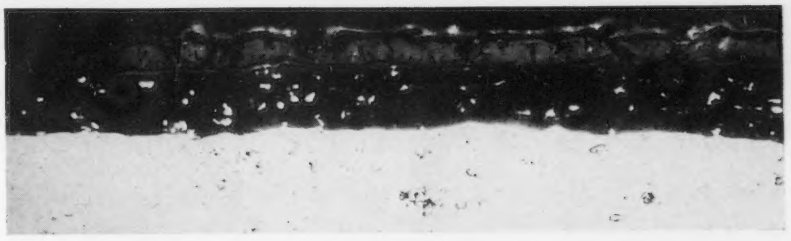
Short term static tests were made on a large number of alloys as a means of selecting for more rigorous testing those alloys showing good corrosion resistance. The results of these tests are summarized in Tables 1 and 2 in which the numerals refer to weight gain in mg/cm² and letters to the types of attack defined above.

In Table 1, alloys low in nickel and copper were susceptible to blistering and pitting, even during exposures as short as 48 hours. An increase in the content of either constituent improved the performance of the alloy. Best results were obtained with compositions in the lower right hand corner of the table. A typical cross section of the corrosion film on the 2 Cu, 2 Ni alloy (6 weeks exposure) is shown in Figure 2a. This should be compared with Figure 1, which shows Type D attack on the 0 Cu, 0.5 Ni alloy after 48 hours.

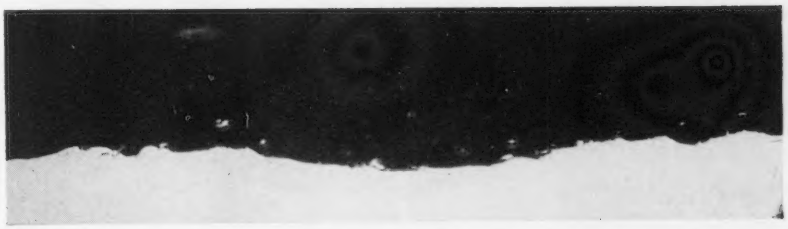
Of this group of alloys, the 2 Cu, 2 Ni alloy was chosen as the most promising, and will be designated in the remainder of this report as Alloy 151. Alloys with higher contents of copper were considered undesirable from the standpoint of limited workability.

Of the Al-Ni-Fe-Si series, it will be seen that the greatest corrosion resistance was exhibited by those alloys whose nominal compositions lie in column 2 of Table 2. These alloys will be designated Alloys 155 and 157. Their actual compositions are listed in Table 3 together with that of Alloy 151. Polished cross sections of corroded specimens of these alloys are shown in Figure 2.

The results summarized in Table 2 show that there is an optimum content of silicon in the neighborhood of 0.2 weight percent. No explanation can be given for this effect, other than to note an apparent relationship between silicon content and the size and distribution of the (Al-Fe-Ni) intermetallic particles. In alloys 155 and 157 the second phase particles were relatively small and finely divided, whereas in the corresponding alloys with 0.5 Si the particles were



A—Alloy 151



B—Alloy 155

C—Alloy 157

Figure 2—Corrosion film on alloys 151, 155 and 157 after six weeks in static water at 300 C. Original magnification of top specimen was 600X (510X after reduction for engraving purposes); original magnification of bottom two specimens was 500X (425X after reduction).

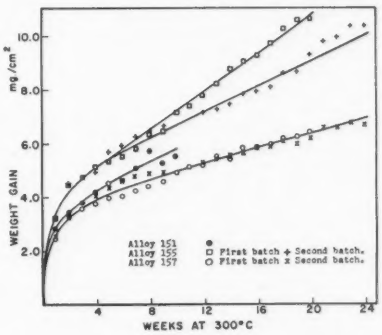


Figure 3—Weight gains (mg/sq cm) of alloys 151, 155 and 157 as a function of time at 300 C.

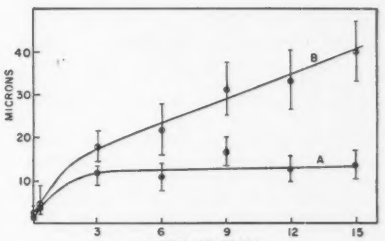


Figure 4—Change in thickness of films A and B on alloy 155 as a function of time at 300 C.

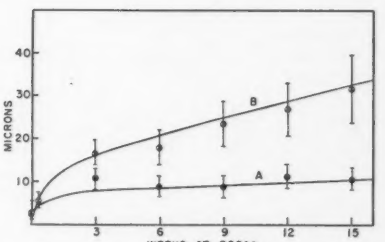
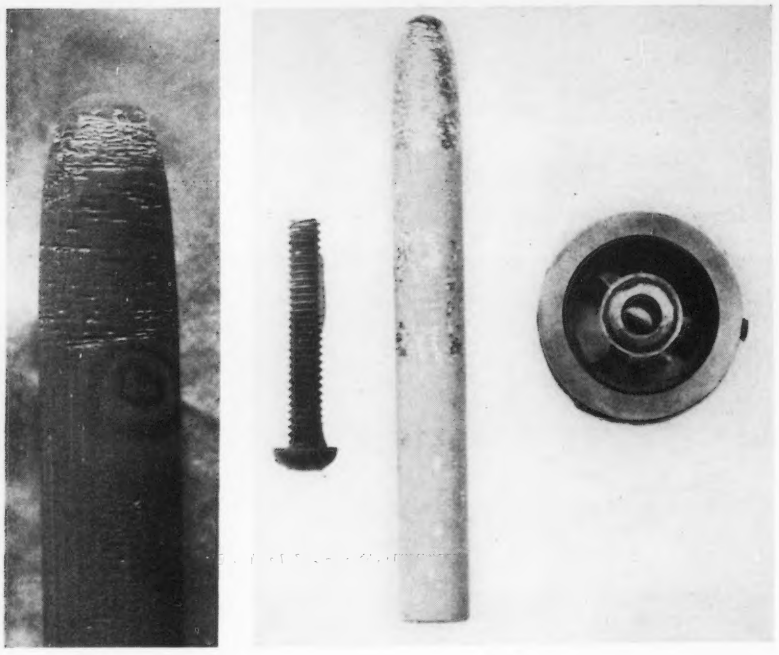


Figure 5—Change in thickness of films A and B on alloy 157 as a function of time at 300 C.

TABLE 2—Summary of One Week Static Tests at 300 C on Al-Ni-Fe-Si Alloys Based on Alcan H.P. Aluminum

Weight Percent Ni	0.5. Fe			1.0 Fe		
	0 Si	0.2 Si	0.5 Si	0 Si	0.2 Si	0.5 Si
0.5	(2.1) C	2.5 A	(2.6) D	(2.3) C	3.0 AB	(2.8) C
2.0	(3.2) C	2.5 A	(3.0) D	2.1 C	2.9 B	2.8 C

Note: Numbers = mg/cm² weight gain; letters = type of attack.



A B

Figure 6—Erosion pattern on solid rod of alloy 155 after 370 hours in deionized water at 260 C flowing at 20 ft./sec. Magnification of A is 2X and that of B is 1X.

TABLE 3—Compositions by Weight Percent of Selected Al-Ni-Cu and Al-Ni-Fe-Si Alloys

ALLOY	Ni	Cu	Fe	Si
151	2.05	2.13	0.25	0.09
155	0.53	0.002	0.49	0.21
157	2.00	0.002	0.48	0.21

TABLE 4—Corrosion Rates of Alloys in Static Water at 300 C

	PENETRATION IN INCHES PER YEAR		
ALLOY	Wt. Gain	Wt. Loss	Film Thickness
151	1.5×10^{-3}	.....	.....
155	2.1	2.4×10^{-3}	2×10^{-3}
157	0.9	0.9	1.3

TABLE 5—Effect of Ionizing Radiation on Corrosion of Al-Ni Alloys

	PENETRATION, INCHES	
ALLOY	In-reactor	Out-of-reactor
151	2.4×10^{-3}	2.7×10^{-3}
155	2.5	2.6
157	2.3	2.7

Intensity of energy absorption~0.1 watt/gm; exposure 700 hours at 260 C (500 F) at 3 ft/sec.

coarse and widely separated. All alloys had similar metallurgical histories.

The results summarized in this section are in general accord with similar results published by Draley and Ruther,[1,2] Huddle and Wilkins[6] and Dillon, Wilson and Troutner.[7] From these combined results it may be concluded that the resistance of aluminum to corrosion by water at temperatures of 250 C-350 C may be improved by:

(a) Addition of copper, which is partly in solid solution and partly as a $CuAl_2$ second phase in the aluminum at the test temperature.

(b) Additions of nickel and iron, which exist only as an insoluble second phase at the test temperature. Reference will be made below to the size and distribution of the second phase particles in these alloys.

Long Term Corrosion Tests

Alloys 151, 155 and 157 were subjected to long term static tests at 300 C in order to determine corrosion rates. Curves showing weight gains expressed as mg/cm² are plotted against the time at 300 C in Figure 3. Each point represents the average weight gain of at least six samples.

Polished cross sections of typical corrosion films at 6 weeks are shown in Figure 2. It will be noted that films on all the specimens have a characteristic double-layered structure. The outer film appears porous. The inner one is evidently denser and more compact, and in contrast to the outer layer, contains particles of the second phase. For convenience in the discussion which follows the outer film will be referred to as Film A and the inner film as Film B.

It is evident from Figure 2a that the second phase particles in Film B have the same size and distribution as in the underlying alloy. A close inspection of this figure shows that Film B is formed by an attack on the metal at the film-metal interface.

A second measurement of the corrosion rates of alloys 155 and 157 was made by measuring the change in the thickness of Films A and B with increasing times of exposure at 300 C. Twenty measurements were made of the thickness of each film, and an arithmetic average thickness determined. The results are plotted in Figures 4 and 5 showing the maximum positive and negative deviations from averages. These curves show that Film A characteristically reaches a limiting thickness after 2-3 weeks at 300 C. Film B continues to grow linearly with time up to 15 weeks. It will be noted that a similar linear increase in weight gain with time was found in Figure 3.

A third measurement of static corrosion rates was obtained by determining the weight lost after film stripping (see page 44). It was found that corrosion rates calculated from weight loss measurements on Alloys 155 and 157 agreed with corrosion rates calculated from weight gains, provided it was assumed that the corrosion product was bŏhmite, $\alpha\ Al_2O_3 \cdot H_2O$. This is the stable modification of the oxide in contact with water at 300 C.[8]

A comparison in Table 4 of corrosion rates calculated from the three essentially independent sets of data shows excellent agreement among the three rates.

These corrosion rates are in general agreement with static rates measured on similar alloys by Draley and Ruther,[1] Dillon, Wilson and Troutner[7] and by Wilkins.[9] The mutual agreement among the three sets of data represented in Table 4 leads to some interesting conclusions:

(a) The weight gain curves are a reliable measurement of static corrosion rates of the resistant alloys (i.e., film losses by flaking, solution, etc., are unimportant.)

(b) This being so, Film B does not appear to be protective, since an increase in the thickness of a protective film would cause a corresponding diminution of the corrosion rate. Film B may, however, be growing by a "crack and heal" mechanism which could result in roughly linear rate of growth. Numerous cracks are in fact found in these films after exposures of 2-3 weeks, but there has been no evidence for enhanced attack on the alloy at these points.

The immediate practical conclusions to be drawn from these results are:

1. The best Al-Ni-Cu and Al-Ni-Fe alloys corrode at acceptably low rates in static water at 300 C.
2. The low corrosion rates are, however, reached only after corrosion films of 10-20 μ thickness have been built up.

This second conclusion has important implications for the dynamic corrosion tests about to be described.

Dynamic Corrosion Tests

In this section reference will be made to tests made on solid cylinders of alloy or alloy tubes with welded end-caps. These samples were mounted in test sections of stainless steel loops in which the linear velocity of flow over the sample was usually 18-20 ft/sec. The most important results of this work are summarized as follows:

1. "Erosion" Corrosion and Mechanical Damage to Films.

It has been the experience of all workers in this field that corrosion rates of

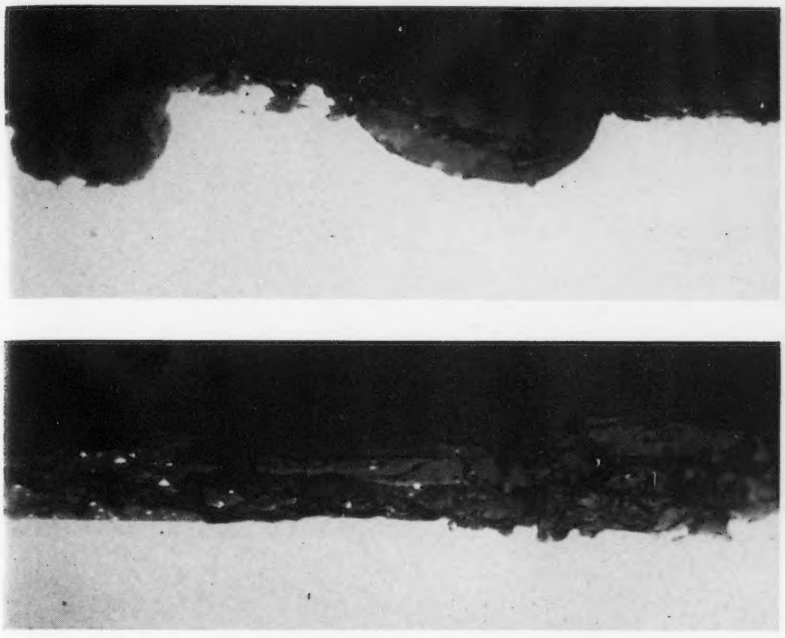


Figure 7—Fragmented film on leading end of sample in Figure 6. Original magnifications were 500X (425X after reduction for engraving purposes).

Figure 8—Sample of seam-welded tubing of alloy 157 (defilmed) after 550 hours exposure to water at 260 C flowing at 18 ft./sec. 2.5X.

Al-Ni alloys are higher in dynamic than in static tests.[1,2,10] Evidence for "erosion" corrosion and mechanical damage to the corrosion film has so far been unique to tests at Chalk River, and there is some lack of agreement between these tests and tests made at United States installations. The discrepancies are now under examination, and it seems possible that differences in test temperatures, details of sample design and mounting, and characteristics of individual loops may all be contributing causes. Meanwhile, the effects to be described are of sufficient practical importance to be given serious consideration.

Evidence for the "erosion" corrosion of alloy 155 is shown in Figure 6. The sample was a bullet-shaped rod, 4.0 inches long x 0.500 inches in diameter, machined from extruded bar stock. It was mounted rigidly in the test section on a spider securely fastened to the pipe conducting water over the test specimen.

After an exposure of 368 hours to deionized water at 260 C (500 F) and 1000 psi flowing with a linear velocity of 20 ft/sec, damage to the leading end of the sample was manifested in a series of erosion rings, beginning a short distance from the tip and extending 15-20 mm along the tapered end. The cylindrical body of the sample was covered with a very smooth, translucent film different in appearance from that produced on alloy 155 in static tests. Subsequent metallographic examination showed that Film A had failed to form or had been scoured from these surfaces by the fast flowing water, leaving only Film B.

The test was continued for a total exposure of 920 hours during which the eroded area increased until it extended half the length of the sample (Figure 6b). Metallographic sections were made of the leading end of the sample, and magnified views of cross sections of the erosion rings are shown in Figure 7. The corrosion film, mainly Film B, has been shattered into fragments at the points of greatest attack. This provides strong evidence for some form of mechanical action of the water.

The mechanism of this type of attack has yet to be resolved. It may be suggested that the damage was caused by vibrations excited in the sample or test section by fundamental vibrations in the loop. The main source of such vibrations was the pump itself, which produced pulsations with a fundamental frequency of 350-400 cps. The damage to a sample with some freedom of vibration can be striking. A typical result is shown in Figure 8 which shows a length of alloy 157 tubing defilmed after an exposure of 550 hours at 260 C (500 F) to water flowing with a linear velocity of 18 ft/sec.

The tubing was fabricated by heliarc seam-welding cold-rolled sheet. At the start of the test the sample was firmly fixed on a stainless steel mandril, but by the end of the test the sample had considerable freedom to rattle. The wrought alloy has been etched away in a distinct pattern of ridges whose periodicity suggests vibration of the sample or the test section at some strong resonance frequency. The spacing of ridges indicates a frequency of the order of 4000 cps, but the existence of resonances at this frequency has yet to be demonstrated.

Whatever the cause of the damage, the susceptibility of the Al-Ni-Fe alloys to this form of attack appears established. Similar effects have been observed with the Al-Ni-Cu alloys. The attack can be minimized by suitable design of sample and test section, but real improvement probably can be achieved only by modifying the characteristics of the alloys. Two desirable modifications are immediately suggested by the results just discussed. The first is to change the alloy or its environment (i.e., with an inhibitor) so that the protective film is thin and less likely to crack and flake. The second is to improve the high temperature strength of the alloy underlying the film. The net result of these modifications would be to diminish the gap between the mechanical properties of the metal and those of the film.

TABLE 6—Details of Specimen Fuel Element

Fuel	Compacted UO_2
Average diameter of compact	0.249″
Average length of compact	0.250″
Total fuel length	3.5″
Sheath	Alloy 151
Average diametrical clearance	0.001″
Maximum diametrical clearance	0.0015″
Axial clearance	0.0″
Average sheath thickness	0.030″
Overall length	4.5″
Heat flux with:	
Natural UO_2	~ 15 watts/cm²
4.7% U_{235}	~ 100 watts/cm²
Calculated sheath surface temperatures:	
Natural UO_2	288 C
4.7% U_{235}	300 C

Because of the accelerating effects of mechanical damage on corrosion it is difficult to establish corrosion rates at water velocities of 18-20 ft/sec. Maximum corrosion rates for freely vibrating samples may be as high as 0.030 inch/month. Rates for flat samples rigidly clamped in the test section have been as low as 0.020 inch/year.

It should be pointed out that the seam weld visible in Figure 8 shows varying degrees of corrosion resistance. The as-cast weld metal has resistance superior to that of the wrought alloy, whereas the transition region between the as-cast and wrought metal has corroded very extensively in this exposure. It is not proposed to discuss corrosion resistance of fusion welds in this paper, but merely to note that fusion welds were found to be unreliable. For making specimen fuel elements a technique of hot pressure welding was developed which avoided the necessity of melting the alloy. Such welds were found to have corrosion resistance equal to that of the wrought metal.

2. Effect of Reactor Radiation on Corrosion.

A comparison was made of the extent of corrosion of identical sets of samples

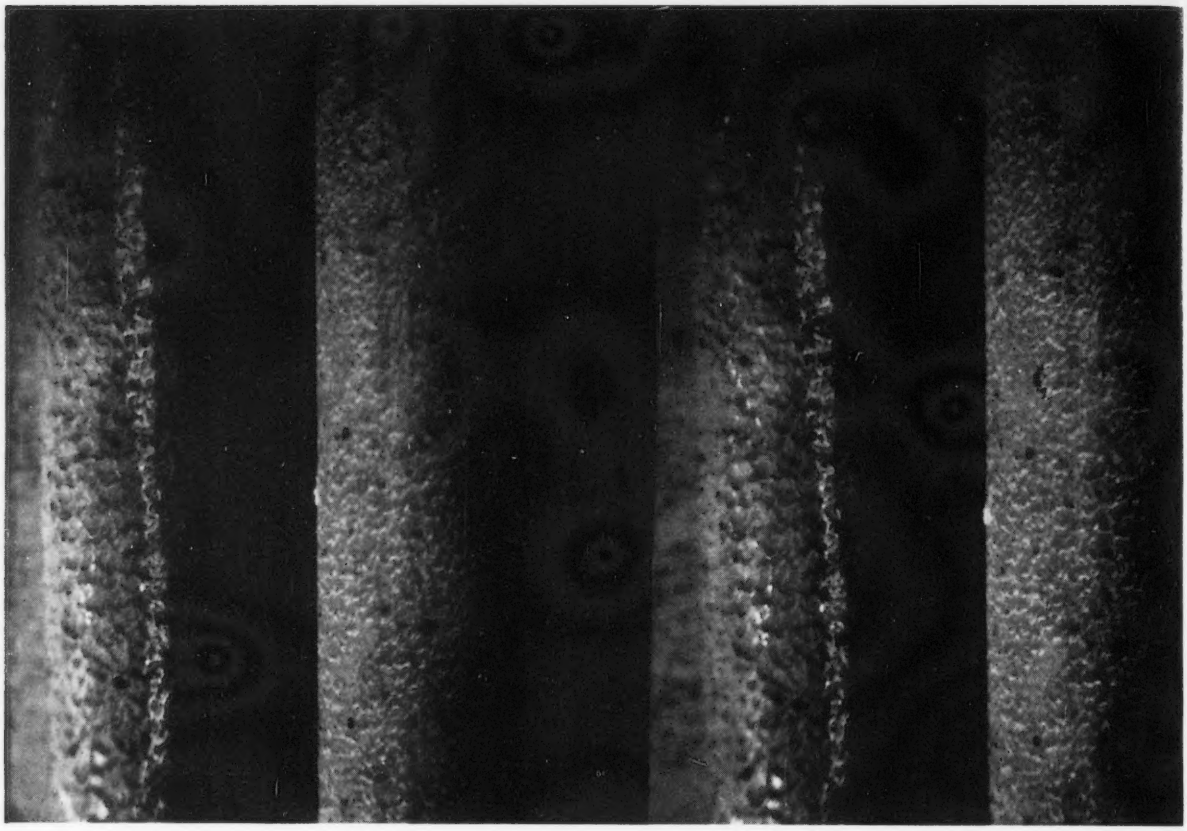


Figure 9—Surface of alloy 151 sheaths after six weeks at 280 C, 18 ft/sec in X-2 Loop. Stereo pair F enriched fuel, C natural fuel. 3.3X.

tested simultaneously in a loop with one test section in an experimental hole in the NRX reactor and a second outside the reactor. The extent of penetration was measured on samples defilmed after an exposure of 700 hours to water at 260 C (500 F) flowing with a linear velocity of 3 ft/sec. The results are shown in Table 5.

Ionizing radiation appears, on the basis of these results, to reduce the penetration of the alloys. It is doubtful whether the reduction was significant, and it may be concluded that radiation of this intensity has a negligible effect on the corrosion rates.

3. Effect of Heat Flux and Radiation on Corrosion.

As a culminating experiment the Al-Ni alloys were tested as actual fuel sheaths on specimen fuel elements in the NRX reactor. Heat fluxes of the order of 100 watt/cm² were obtained by using enriched UO_2 as a fuel core. The details of a typical test specimen are given in Table 6.

The completed fuel samples were fitted with Type 304 stainless steel adapters which enabled several samples to be connected together, and at the same time to be spaced from the walls of the tube forming the test section.

Conditions of exposure were 288 C (550 F), 2000 psi static pressure and a linear flow velocity of 18 ft/sec. The water was deionized by mixed bed ion exchange resins.

Figure 9 shows a typical sample of 151 with ~ 100 watts/cm² heat flux after six weeks exposure. Corrosion rates measured in this interval indicated a penetration of roughly 0.010 inch/month. A slight acceleration of attack was noted in samples containing the enriched UO_2, a sevenfold increase in heat flux producing an approximate 10 percent increase in corrosion rate. The most important effect was, once again, mechanical damage to the corrosion film.

A suitably rigid mounting for samples of this kind has yet to be devised, and what the corrosion rate would be under ideal conditions can only be a matter for conjecture. That radiation and heat flux have relatively minor roles in the corrosion of the aluminum-nickel alloys at high temperatures and flow velocities seems established.

Summary

The conclusions drawn from the experimental work just described may be recapitulated briefly as follows:

As demonstrated by Draley[1] and others,[6,7,9] aluminum can be made resistant to high temperature water by small additions of Ni, Fe and Cu. The second phase particles of these alloys must be small and evenly distributed for optimum corrosion resistance. In the presence of an adequate concentration of second phase, the high temperature attack changes from localized penetration at grain boundaries and blister formation to more desirable uniform overall attack. The latter causes two films to be formed, Film A growing outward from the original metal surface and Film B growing inward into the metal. Film A reaches a limiting thickness after 2-3 weeks at 300 C, but Film B continues to grow linearly with time. At this stage the corrosion rate is acceptably low ($\sim 1 \times 10^{-3}$ inches/year) but the corrosion film is 10-20 μ thick, relatively brittle and subject to severe mechanical damage in dynamic tests. Reactor radiation has an insignificant effect upon corrosion. Heat fluxes of the order of 100 watts/cm² also appear to have little effect, though in this instance the results may have been obscured by mechanical damage.

Clearly, the greatest obstacle to the use of Al-Ni alloys in their present state of development is the friability of the corrosion film. Coupled with this are the relatively poor mechanical properties of the alloys at elevated temperatures. An improvement must be made both in the direction of diminishing the thickness of the protective film and increasing the

strength of the alloy at working temperatures.

Acknowledgments

The results described in this paper are the end product of the labors of several groups and individuals both at Chalk River and elsewhere. Work on aluminum-nickel was undertaken at Chalk River chiefly at the instigation of E. C. W. Perryman, whose continued help and interest are gratefully acknowledged. Much of the work described was done by G. J. Biefer, J. S. Nelles and others at Chalk River. The alloys were prepared by Aluminium Laboratories, Ltd., Kingston, Ontario. Tubing and extruded bar were fabricated at the Mines Branch, Ottawa.

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NACE TECHNICAL COMMITTEE REPORT

Corrosion of Aluminum In High Purity Water*

By R. J. LOBSINGER[1] and J. M. ATWOOD[2]

Abstract

In-reactor corrosion rates of 1245 and M-388 aluminum alloys were determined in demineralized water at temperatures up to 200 C. A correlation between corrosion rates and the effective aluminum surface temperature is presented. The effect of increased hydrogen ion concentration on corrosion rates is also discussed. 4.6.1

Introduction

THE USE of an aluminum base alloy in nuclear reactors is desirable because of ease of fabrication, relative low cost, and low neutron cross section. The upper temperature at which aluminum can be used in a water coolant system appears to be limited by corrosion effects associated with increasing temperature.

In order to study aluminum corrosion problems in a recirculation system, a test loop was operated at Hanford from November 1952 to October 1956. The testing was performed to establish the corrosion rates obtained in demineralized water at temperatures up to 200 C. In addition, a recently developed aluminum alloy was tested in an effort to obtain better corrosion resistance at these conditions. The effect of increased hydrogen ion concentration on corrosion was studied as a part of the program.

This report presents the corrosion rates obtained on 1245 and M-388 aluminum alloys tested in the recirculation system.

Description of Equipment

A schematic diagram of the test facility is shown in Figure 1. Steam condensate is deoxygenated and then deionized to obtain a purity of 0.1 ppm total dissolved solids. This water is recirculated through the loop system by a series arrangement of three pumps. The water is cooled, depressurized, and

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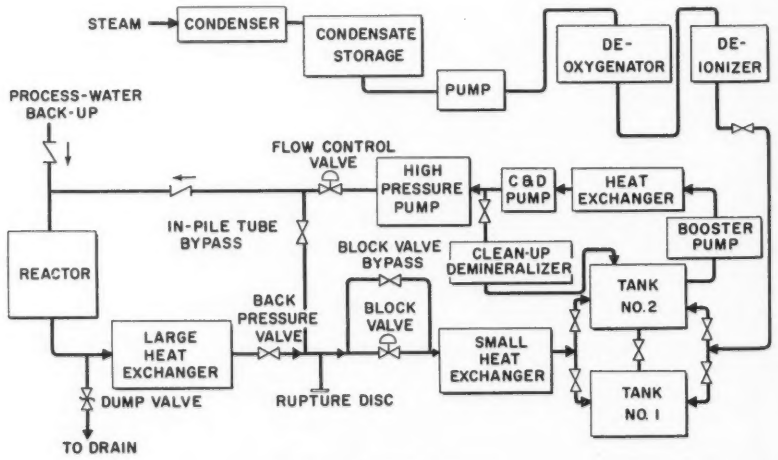

Figure 1—H-Loop flow diagram.

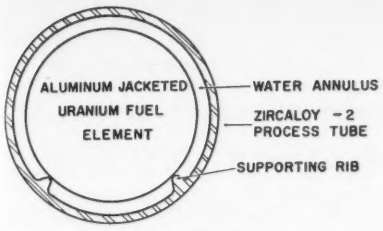

Figure 2—Transverse cross-section of process tube showing fuel element resting on ribs.

sent to retention tanks before being recycled. During normal operation a retention of one to two hours served to reduce the activity by a factor of 10. A small by-pass deionizer unit was used to maintain the system water quality. In tests where high purity water was desired, a small portion of the main stream was sent through the unit which had been filled with a mixed-bed ion exchange resin. This removed any corrosion products picked up by the water.

For low pH testing, a cation exchange resin was used in the unit. The use of this cation resin permitted obtaining pH values in the range 5.3-5.6 as compared to values of 5.8-6.2 observed with ordinary mixed-bed clean-up. Still lower values of 4.5-5.0 were obtained by direct phosphoric acid injection.

The test section consisted of a Zircaloy-2 tube which had two longitudinal ribs for support of the samples. Figure 2 shows a cross section of the tube with a sample resting on the ribs.

Procedures

The corrosion rates of the aluminum samples were obtained by weight loss measurements after removal of the corrosion product. The samples were then cleaned in a chromic acid-phosphoric acid solution. The weights obtained after cleaning and drying gave the amount of aluminum lost and permitted calculation of a corrosion rate.

Discussions and Results

Initial testing in the facility was performed using high purity water. Several tests on 1245 alloy were made at varying periods of time. In these tests, samples were tested both in the presence and absence of heat transfer. It was found that in the absence of heat transfer, rates were consistently lower. The corroding surface temperature in the absence of heat transfer was essentially equivalent to the local water temperature; however, where heat transfer was present the corroding surface temperature was significantly higher than the local water temperature.

The corrosion rates obtained in the absence of heat transfer were plotted as a function of surface temperature. Then it was determined for heat transfer conditions at each corrosion rate what ΔT due to heat transfer should be added to the water temperature to obtain a corroding surface temperature predicted from the data obtained in the absence of heat transfer. These factors were averaged and the average value was used to calculate an effective surface temperature. This correlation was obtained by

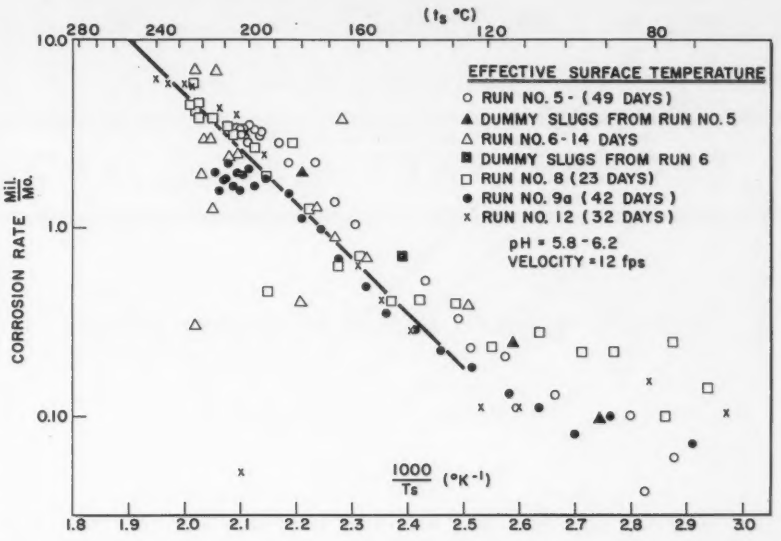
(t_s °C)
280 240 200 160 120 80
10.0
1.0
0.10
CORROSION RATE Mil/Mo
EFFECTIVE SURFACE TEMPERATURE
RUN NO. 5 - (49 DAYS)
DUMMY SLUGS FROM RUN NO. 5
RUN NO. 6 - 14 DAYS
DUMMY SLUGS FROM RUN 6
RUN NO. 8 (23 DAYS)
RUN NO. 9a (42 DAYS)
RUN NO. 12 (32 DAYS)
pH = 5.8 - 6.2
VELOCITY = 12 fps
1000/Ts (°K⁻¹)
1.8 1.9 2.0 2.1 2.2 2.3 2.4 2.5 2.6 2.7 2.8 2.9 3.0

Figure 3—Aluminum corrosion rate versus the reciprocal of the effective surface temperature. pH was 5.8-6.2.

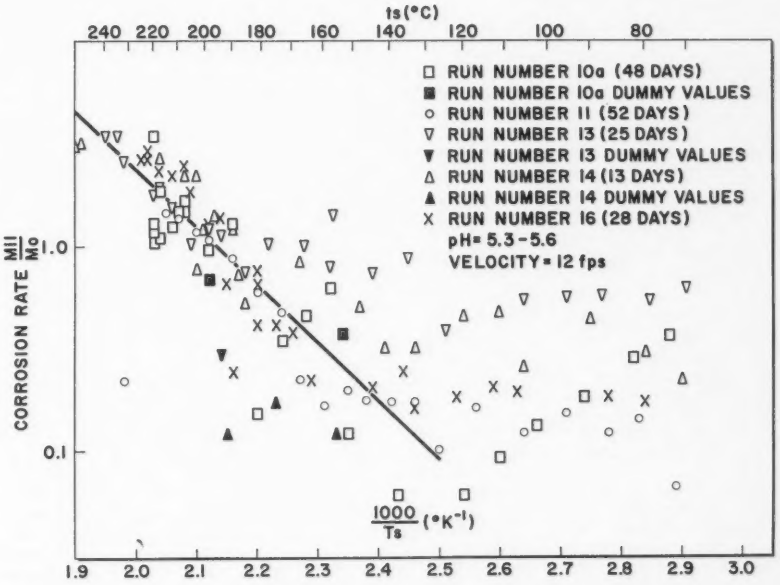
ts(°C)
240 220 200 180 160 140 120 100 80
1.0
0.1
CORROSION RATE Mil/Mo
RUN NUMBER 10a (48 DAYS)
RUN NUMBER 10a DUMMY VALUES
RUN NUMBER 11 (52 DAYS)
RUN NUMBER 13 (25 DAYS)
RUN NUMBER 13 DUMMY VALUES
RUN NUMBER 14 (13 DAYS)
RUN NUMBER 14 DUMMY VALUES
RUN NUMBER 16 (28 DAYS)
pH = 5.3 - 5.6
VELOCITY = 12 fps
1000/Ts (°K⁻¹)
1.9 2.0 2.1 2.2 2.3 2.4 2.5 2.6 2.7 2.8 2.9 3.0

Figure 4—Aluminum corrosion rate versus the reciprocal of the effective surface temperature. pH was 5.3-5.6.

N. R. Miller and the authors, and a plot of corrosion rates versus the reciprocal of the effective surface temperature thus obtained is shown in Figure 3. This correlation holds reasonably well down to approximately 125 C. At lower temperatures scatter is obtained. This scatter may be due to larger relative weighing errors at these lower temperatures. Also, a different form of corrosion product may be produced changing the effect of heat transfer.

Draley[4] has obtained similar breaks in his corrosion rate versus temperature curves. Several tests were made on alloy 1245 at pH values from 5.3-5.6 to determine the effect of hydrogen ion concentration on aluminum corrosion. The results of these tests are shown in Figure 4. The reduction in pH reduced corrosion by a factor of about two. Again, as in the high purity data, the correlation is valid down to about 125 C.

Testing was performed in later phases of the program using M-388 alloy to prevent intergranular corrosion. One high purity run was made with this alloy and is also shown on Figure 3. No apparent difference is noted in the overall corrosion rates of 1245 or M-388

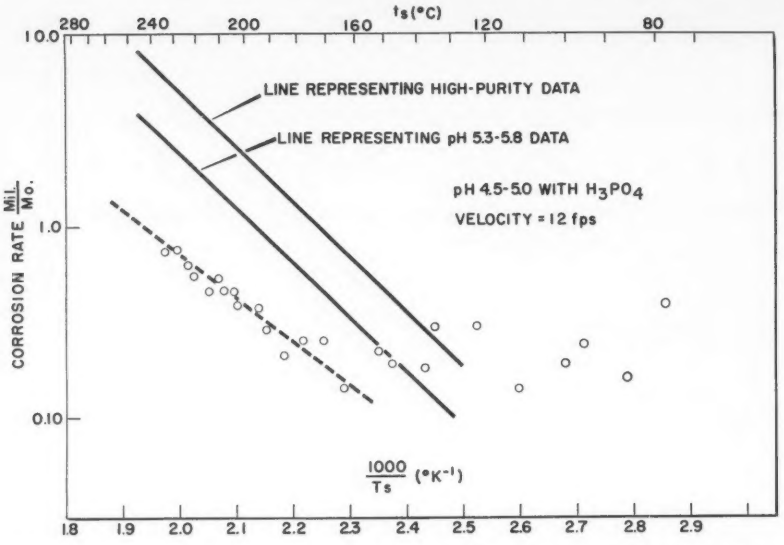

Figure 5—Aluminum corrosion rate versus the reciprocal of the effective surface temperature. pH was 4.5-5.0 with H_3PO_4.

alloys. Similarly, in the low pH studies no difference in corrosion rates was found.

In an effort to lower corrosion rates still further, a final run was performed in the facility using injected phosphoric acid to reduce the pH to 4.5-5.0. These data are shown in Figure 5. As can be seen, a marked reduction was obtained. Compared to high purity water, the rates were reduced by a factor of approximately seven.

Conclusions

The testing performed in the loop led to the following conclusions:

1. M-388 and 1245 aluminum alloys have comparable overall corrosion rates.
2. The corrosion rate of the alloys can be reduced substantially by reducing the pH.

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NACE TECHNICAL COMMITTEE REPORT

The Storage of High Purity Water*

By RICHARD R. DLESK*

Abstract

A need is developing in industry for water of higher and higher purity. As the purity of the water increases, the storing of it becomes an increasingly difficult problem. In order to mitigate this problem, investigations and appraisals of new protective measures for existing steel tanks were made, and some were applied to actual installations. Protective coatings discussed include red lead paint, metallic zinc paint, synthetic rubber, nickel-phosphorous-alloy coating, heavy electroplated nickel, and metallized aluminum coatings.

Aluminum, as a construction material for storage facilities, was evaluated in laboratory and field tests. These tests resulted in the construction of four 44,000-gallon aluminum storage tanks. Data were reported to show the electrical resistance of a film developed on 3003 aluminum alloy in tap water, distilled water and demineralized water.

4.6.5

Introduction

MODERN STEAM generators operate at temperatures and pressures which were only a dream a few short years ago. As temperatures and pressures have steadily increased, there necessarily has been an accompanying demand for water of higher and higher purity. This need for high-purity water has bred a multiplicity of complex problems. The economical storage of this water is one of these problems.

High-Purity Water

The term "high-purity water" has about as many connotations as there are different uses for water. The public health official, the manufacturer, the brewer, and probably even the candle stick maker each has his own idea as to what constitutes a water of high purity. Of course, "high-purity," or to be more correct "highest-purity," water can be defined as a water free of all solids, liquids, and gases; however from a practical standpoint this definition is not valid.

Generally, the electric utility industry defines high-purity water as a water low in hardness, silica, and dissolved gases, with an electrical resistance of approximately 500,000 ohm-cm. However, "high-purity" is not a static concept. The high-purity water of today is markedly different from that of ten years ago, or even five; what was "high-purity" in 1952 is "impure" today. Tomorrow will bring even greater changes. The nuclear generation of power will require a water having an electrical resistance of from 1 to 10 million ohm-cm.

The Storage Problem

Unfortunately, the increase in purity of the water used in steam generation has been accompanied by an almost proportional increase in the corrosion rate of steel water-storage tanks. This increase, which is serious from both economical and maintenance-of-purity reasons, necessitated an intensification in the efforts being made to mitigate or, preferably, to eliminate the corrosion.

The problem has two general aspects: (1) the economical protection of existing steel water-storage facilities, and (2) the need for a moderately priced, corrosion-resistant container.

Protective Coatings

Several types of coatings, which seemed applicable for protecting existing water-storage facilities from the deleterious effects of the high-purity water, were investigated in the laboratory. Some coatings were applied to field installations, with varying degrees of success.

On the whole, the protection of water-storage tanks with various types of coatings has not been too effective. The successful application of most coatings is limited by several factors: (1) the temperature of the water, (2) the porosity of most coatings, and (3) the possibility of contaminating the water. The maintenance of the continuity of the coating on the submerged portions of the tanks is also extremely important. The tanks are subjected to an oxygen-concentration cell type of attack, which would tend to concentrate the corrosion at the breaks in the coating, with the possibility of early perforations instead of an evenly corroded total surface.

Red Lead Paint

Laboratory tests were made on two different types of red lead paint: Lead Industries Association Formula 7-4, a red-lead-silicates pigment in a phenolic varnish vehicle, and Formula 7-1, a red-lead pigment in a linseed oil vehicle.

The interior surfaces of four steel cylinders were sand blasted and then

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sprayed with paint before new rusting could begin; two cylinders were sprayed with each kind of paint. After air-drying at 85 F for 72 hours, the paint film on one of each pair of cylinders was intentionally damaged by scratching with a blunt instrument. The four cylinders were then filled with distilled water. After one week and four weeks contact with distilled water at 85 F, the cylinders were examined and the amount of silica in the water was determined. (Silica is very undesirable in high-pressure-boiler water. If present in a concentration of over 1 ppm, it may be carried with the steam into the turbine and deposited on the turbine blading. These deposits greatly decrease the efficiency of the machine.)

The tests indicated that Formula 7-1 was the better formulation for the protection of storage tanks. The steel surface under the damaged areas painted with Formula 7-1, the linseed oil paint, was not rusted after four weeks, while the steel under the scratched areas of the phenolic varnish paint rusted in less than a week.

The silica pick-up from the 7-4 formula was only 0.05 ppm, which is within the acceptable limit; however, in view of the ever-present possibility of a major failure of the coating, with large areas of the red-lead film sloughing off, Formula 7-4 paint could put large amounts of the undesirable silica into the boiler water. Application of red lead-linseed oil paint to actual installations has shown it probably to be the most promising economical coating for effectively combating corrosion in low-temperature storage facilities. However it is far from being the ultimate in perfection. This imperfect efficacy is illustrated by the case of a 400,000-gallon steel tank for the storage of demineralized water which was installed in September 1953. Prior to construction of the tank, the fabricated carbon-steel sheets received a pickling treatment and while still warm were given a brushed shop coat of red lead in a linseed oil vehicle (the paint pigment also contained some barytes, iron oxide, and graphite). After erection, all bare spots were touched up in the field with red-lead primer. Between September 30, and November 30, three coats of linseed oil red-lead paint were applied. After two years, the tank was emptied and inspected.

Numerous small blisters were present in large patches on about 20 percent of the surfaces of the tank, with the areas in between in good condition. The larger blisters were about ½ inch in diameter. A few of the blisters contained water, the rest air. Those with water broke under pressure; those with air tended to lift the adjacent paint film. The blisters which broke showed the shop-applied prime coat to be intact, which indicated poor adhesion of the field coats to the prime coat. The coating was in perfect condition at all welds, where the shop coat had been burned off and then touched up with red lead primer in the field. It is believed that the primer and finish formulation were compatible; however, it is possible that the undercoat was overcured, which could cause poor between-coat adhesion.

Metallic Zinc Paint

Metallic zinc paint was used as a protective coating with spectacularly unsuccessful results. Before two 30,000-gallon steel tanks were placed into demineralized-water storage service, the fabricated steel sheets were given a shop coat of metallic-zinc paint. Two additional coats were applied in the field after erection of the tanks.

After five years of service, an inspection was made of the inside of the tanks. The coating had completely failed sometime previously. On the side walls, there were large rust tubercles with deep pits underneath. The floor was covered with two inches of red rust mixed with a gray chalked muck, which had once been the "protective" paint coating.

Synthetic Rubber

Laboratory tests and field experience have shown that synthetic rubber coatings do not give adequate protection to distilled water tanks.

Two steel panels, coated with one dipped coat of primer and ten dipped coats of synthetic rubber, were fastened in a tank containing distilled water at a temperature between 160 and 180 F. After an exposure of 21 months the coatings had become very brittle and were alligatored so badly that some cracks penetrated through the coating to the underlying metal. Additional samples, tested in distilled water at 150 F were badly blistered after 11 months.

During December 1949, a 100,000-gallon tank was sandblasted and after application of a primer and accelerator, 11 coats of rubber coating were rolled on to a thickness of 1/32 inch. Due to the low outside temperature, it was necessary to blow warm air into the tank in order to apply the paint properly.

An inspection one year later showed that the coating had blistered badly. The blisters were small, the largest being about ½ inch in diameter. After an additional three years of service, the condition had worsened. The blisters were more numerous and seemed more brittle. Many were filled with rust-colored water.

Another tank at the same location was painted during July 1950. The same procedure was used, except, of course, the warm air was not needed. The coating on this tank, unfortunately, duplicated the failure of the other coating.

Nickel-Phosphorous-Alloy Coated Steel

Three nickel-phosphorous alloy-coated steel panels were immersed to approximately 5/8 their length in distilled water at 150 F, and insulated electrically to prevent electrolysis. Two of the test panels were coated to a thickness of 3 mils, one to a thickness of 1 mil. After one month's immersion, each of the panels contained small rust spots and rust tubercles.

Heavy Electroplated Nickel

Preliminary tests indicate that a coating of heavy electroplated nickel shows promise as a protective coating for steel. Steel specimens, coated with 8 mils of electrodeposited nickel have been immersed in oxygen-saturated distilled water at 130 F for six months. The specimens seem to be entirely pore-free as evidenced by the absence of pin-hole rusting or bleeding through the coating. It is understood that such coatings can be deposited before tank fabrication, and that welding the coated side with nickel rod, and back-filling the underside with carbon steel ensures a continuous nickel coating. The coating is said to cost in the neighborhood of $1.50 per square foot, which is not cheap.

Metallized Aluminum Coatings

In 1947, two 34,500-gallon distill[ed] water storage tanks were sandblast[ed] and sprayed with metallic aluminum [to] an average thickness of 6 mils, with [a] minimum thickness of 4.5 mils. In 19[52] after more than five years service, [an] inspection showed the coatings on bo[th] tanks to be failing on the submerg[ed] surfaces, except at the overlaps, wh[ere] apparently a thicker layer had been a[p]plied. Water temperatures in these tan[ks] varied from 140 to 180 F.

After the inspection, the tanks we[re] sandblasted and sprayed with metal[lic] aluminum with a supposed nomin[al] average of 8 mils and a maximum thic[k]ness of 12 mils so as to avoid any diffi[-]culties that might be caused by a differ[-]ence in the thermal expansion coefficien[t] of the bonded metals.

After two years of service, an inspection was made of the inside of the tanks. This inspection revealed that in one of the tanks, Tank A:

1. About 80 percent of the submerged areas of one of the tanks appeared to be in good condition. Checks with a magnetic thickness gauge showed a thickness of from 12 to 22 mils in the good areas.
2. In about 15 percent of the submerged side wall areas there were scattered large patches where blistering had occurred. Some of these blisters were as much as 3 inches in diameter and raised ¼ inch from the wall at the center of the blister. They appeared to be hollow, but it was not considered advisable to break one in order to determine the condition of the steel underneath a blister.
3. There were six areas, totaling about 5 percent of the side walls, in which failure had occurred. In these areas, the walls were covered with heavy red rust as much as ¼ inch thick. Numerous flecks of aluminum were visible in the rust. Thickness checks in two of these areas showed from 1 to 1.5 mils of coating material.

During the coating application there was considerable moisture vapor in Tank A, so much so that calcium chloride was brought in to reduce the humidity. The high humidity would quickly cause rusting of the sand-blasted steel and would seriously reduce the adhesion of the applied coating. In the blistered area it is possible that the coating was too thick for the temperature variations that are encountered in this tank.

The metallized aluminum coating in the other reconditioned tank, Tank B, was found to be in far better condition. There were several scattered areas, estimated to be less than ½ percent of the total submerged surface, where corrosion of the steel had occurred through the pores of the metallic aluminum coating. Numerous small mounds of rust had been formed which could be washed off with a wet rag. When cleaned, thickness checks in these areas showed from 4 to 8 mils of coating material, but the surface appeared porous with tiny pinholes from which flecks of metallic aluminum covered with a heavy film of hydrated aluminum oxide could be

picked out with a knife. A few hollow blisters were scattered around the sidewalls of the tank. In the large areas that were in excellent condition, the surface was a uniform dull gray in color and covered with a coating which was from 8 to 12 mils thick.

Based upon field experiences and upon supplemental laboratory tests, a metallized aluminum coating seems to be one of the better surface applications for steel in distilled water service at elevated temperatures. Recent developments such as carbon steel flashing of the sandblasted surface have greatly improved the adhesion of the metallized aluminum to the steel. However, metallized aluminum coatings are expensive. The cost of sandblasting, carbon-steel flashing, and spraying a tank with 12 mils of aluminum is estimated to be about 40 percent of the cost of replacing the tank with one of solid aluminum. This high cost of application together with a coating life of only about 8 to 10 years offsets the superior protective qualities of the coating.

Aluminum Storage Tanks

An aluminum water-storage tank would seem to advantageously combine superior corrosion resistance with economic feasibility. This combination pointed to the construction of solid aluminum tanks to provide needed additional demineralized-water storage facilities.

Although the path leading to the probable solution of the high-purity-water storage problem was pointed out, several possible road blocks needed investigation. The tanks would be filled with water having an initial pH of 9.0 to 9.5 and a concentration of 0.5 to 1.5 ppm of sodium hydroxide, which increased the possibility of serious corrosion of the aluminum. It was also important to determine the amount of aluminum which would be dissolved from the tank. Aluminum is a very undesirable constituent of high-pressure-boiler water. It combines with silica, if the silica is present in high concentrations, to form an extremely hard, difficult-to-remove scale on the boiler tubes.

In order to determine the magnitude of these two possible trouble makers, two 55-gallon drums, fabricated from 3003 aluminum (98.8 percent aluminum, 1.2 percent manganese) were used to simulate the proposed tanks. After the drums were received from the manufacturer, they were conditioned in three steps:

1. Filled with tap water for 24 hours.
2. Drained, flushed and re-filled with tap water for a second 24-hour period.
3. Drained, flushed, and re-filled with distilled water for another 24-hour period.

After this treatment, one drum was filled with reservoir water (part distilled and part demineralized, initial pH 9.0) and the other drum was filled directly from a demineralizer, initial pH 9.5. The drums were completely filled and sealed.

After 18 months, the aluminum content of both waters was determined. It was low, being only 0.03 ppm in the water from each drum. The drum containing the reservoir water was sectioned and found to be in good condition; there was no corrosion, sludge formation, or corrosion product. The interior of the barrel had become coated with a yellowish-white, adherent film of hydrated aluminum oxide.

TABLE 1—Resistance of Oxide Film in Three Types of Water

TIME	RESISTANCE OF OXIDE FILM IN OHMS		
	Tap Water	Distilled Water	Demineralized Water
Initial	0.175	0.175	0.175
One day	0.20	0.19	0.20
One week	0.21	300,000	260,000
Three weeks	0.20	750,000	Greater than 1 megohm
One month	0.28	Infinite	Infinite

When this information was made known to management, a logical step seemed to be to erect some full-size tanks. Satisfactory bids were received and the contract awarded for the construction of four 44,000-gallon tanks to be fabricated from 3003 aluminum alloy.

As the service date approached schedules became tighter, and the possibility of eliminating part or all of the conditioning treatment applied to the experimental barrels arose. Some skepticism of the recommended treatment had been expressed because it seemed to be entirely empirical, and no sound scientific reasons could be advanced to support it. The desideratum was, of course, to obtain a continuous hydrated aluminum oxide coating on the tank surface. Some time was available for exploring conditioning methods, so some panels of 3003 alloy were obtained and immersed in tap water, distilled water, and demineralized water at ambient laboratory temperature. The development of the protective oxide coating was followed by means of resistance measurements.

The measurements reported in Table 1 show that a film having high electrical resistance will develop on 3003 aluminum alloy in distilled or demineralized water in from one to three weeks. The hocus pocus of emptying, flushing, and re-filling the tanks seemed not necessary. The tests also showed that Lake Michigan (tap) water was not a good conditioning agent. Some superficial attack of the aluminum seems to be concomitant with the rapid formation of a high resistance film. Later tests showed that extremely pure demineralized water (as from a mixed bed demineralizer) was a less rapid film former than water containing a part per million of ammonia. Four parts per million of morpholine was less effective than a part per million of ammonia.

During August, 1955, four 44,000-gallon tanks of 3003 aluminum alloy were put into service for the storage of demineralized water. It was decided to condition the tanks by filling them and keeping them filled as completely as possible. To determine aluminum pick-up, one tank was filled and isolated for two weeks. At the end of this time, the water contained 0.04 ppm of aluminum.

After three months of service, an inspection was made of the interior of one of the tanks. The inspection revealed that:

1. The surfaces were in excellent condition. There was no discoloration even at the water line, and only a few small areas of surface action due to impurities spattered on or abraded into the surface during construction, such as small iron turnings which, of course, had rusted. There were some shallow pits on the overflow pipe largely in the portion near the floor. The metal of the overflow pipe appeared to be of a different texture and finish than the floor, although both were 3003 alloy.
2. There were several areas where there was mechanical damage which occurred during erection. These were largely in the nature of clamping tool dents and tack welds. Although such defects are more susceptible to corrosion, none of the damaged areas showed evidence of corrosive action.
3. Some of the weld seams (approximately 20 percent) were dark gray and were slightly pitted. The remainder of the weld seams were light gray and in excellent condition. Samples chiseled off the welds and subjected to spectrographic analysis showed that the light gray weld seams were 1100 aluminum as they should be, whereas the dark gray pitted welds were 4043 silicon-aluminum alloy.

Another inspection was made of these tanks after they had been in service for a year. The surfaces of the tanks were still in excellent condition and the weld seams showed no evidence of further corrosive action.

These aluminum tanks are equipped with steam heating coils, as it has been the practice at this station to maintain make-up water at 120 F or higher to promote de-aeration when the make-up flows into the condenser hot-well. The coil in each tank consists of a ring of 4-inch aluminum pipe (3003 alloy) spaced about 18 inches from the wall and about two feet from the bottom of the reservoir. This pipe ring is connected to the steam supply at its midpoint by a vertical riser and is supported by tack-welding 1-inch aluminum pipe legs to it and to the tank bottom. The four-inch pipe ring is perforated with ½-inch holes by means of which low pressure steam is injected directly into the demineralized water.

Evidently, a steam hammer had developed in No. 4 reservoir, and the steam heating ring and connecting part of the riser were broken into several fragments. Examination showed severe pitting on the steam side (inside) of the ring, evidently from cavitation resulting from collapse of steam bubbles. There was thinning and bulging of the pipe. Similar damage has not been observed in the other three reservoirs, but when de-watered, it is expected that it will be found that the steam injection piping system has been deteriorated by cavitation.

The steam injected into these reservoirs is treated with morpholine to a pH of 9, and some concern developed that this high pH might be damaging to the aluminum. Accordingly, daily pH checks were run for 10 days while steam was being injected to maintain a temperature of 120 F, and for 10 days while no steam was injected. pH was the same during both periods, varying from 6.9 to 7.1. Evidently the quantity of morpholine injected with the steam is insignificant.

To date the performance of these tanks has been satisfactory. It is a gratifying experience to enter one of these reservoirs after draining and note the cleanliness, in contrast to the one or two inches of iron oxide much which is usually found in a steel tank. The reduction in the quantity of iron rust transported to the boiler system is, in itself, worthwhile. Another bonus is the fact that aluminum tanks can be placed outside without the necessity of painting them periodically.

Water Conditions for High Pressure Boilers*

By D. E. VOYLES[1] and E. C. FISS[2]

Abstract

Components of a high pressure boiler system which are in contact with high purity water are described. Materials of construction and operating pressures and temperatures are given for such items as condensers, feedwater heaters and boiler components. Demineralized and evaporated make-up are referred to along with data on the chemical nature of the water. Analyses of water and steam samples from points in the boiler water cycle show iron and copper contents and other indications of the purity and corrosiveness of the water. The part played by chemical treatment, boiler blowdown, removal of dissolved gases and other operating factors in maintaining the described water conditions are discussed. 4.6.2

Introduction

THE PURPOSE of this paper is to outline the experience and practices of the Duke Power Company with regard to water conditions in steam boilers operating in the pressure range of 1350 to 1850 psig with boiler water containing solids of less than 10 ppm.

The installations referred to are similar to the 1850 psig units where steam leaves the boiler at the saturation temperature of about 625 F and is superheated to 1000 F. This superheated steam is expanded through eight high pressure stages of the turbine to 410 psig and returns to the reheat section of the boiler at this pressure where it is heated again to 1000 F. The reheated steam is then expanded through the remaining stages of the turbine and exhausted to the main condenser at an absolute pressure of about 1.5 inches of mercury. The exhaust steam is condensed at approximately 90 F and pumped at 70 psig through two tubular heat exchangers and one open deaerating heater so that the temperature at the suction of the boiler feed pump is approximately 310 F. The boiler feed pump then pumps at full boiler pressure through two additional tubular heat exchangers which gives the feedwater a temperature of about 450 F at the point where it enters the economizer section of the boiler.

Materials of Construction

A brief description of the materials of construction for the equipment in a water-steam cycle such as the one just described is given below.

The boiler tubes and the low temperature sections of the superheaters are of A-192 low carbon steel construction. Tubes in the high temperature section of the superheater and the reheater are of chrome molybdenum steel (A213-T11 and A213-T22). Some of the high temperature stage components of the turbine are constructed of 12 percent chrome steel, the remaining stages being primarily of low alloy or carbon steel.

The tubes of the condenser and the low temperature feedwater heaters are of Admiralty brass and the shells are of plain carbon steel. The condensate pumps have bronze impellers and the boiler feed pump impellers are of 12 percent chrome steel. The tubes of the high temperature feedwater heaters are of 80-20 cupro-nickel and the feedwater piping is of A-106 carbon steel.

Boiler blowdown, venting of the deaerating heater, furnace slag blowing, etc. account for a water loss equivalent to about 0.5 percent of the steam generating rate. This loss is made up by evaporating or demineralizing equipment which is guaranteed to furnish water with total dissolved solids of less than 1 ppm and dissolved silica of less than 0.1 ppm. In the case of evaporators, they are preceded by a phosphoric acid treatment and open feedwater heaters which reduce the dissolved oxygen to less than 0.01 ppm and the total CO_2 to about 0.5 ppm.

The air removal section of the condenser is effective in maintaining a typical dissolved oxygen content of about 0.01 in the condensate and the deaerating feedwater heater is guaranteed to reduce dissolved oxygen to less than 0.007 ppm.

Operating Conditions

An outline of typical operating conditions which relate to the water conditions in these units is given below.

The evaporated or demineralized make-up is about 6,000 pounds per hour or approximately 0.5 percent. The deaerating heater in the feedwater system is vented at 600 to 800 pounds per hour to ensure best possible mechanical deaeration of the feedwater. Condensers

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TABLE 1

	Condensate	Evaporated Make Up	Boiler Feedwater	Boiler Water	Superheated Steam
pH	8.5—9.0	6.0—7.0	8.5—9.0	8.5—9.0	8.5—9.0
Conductivity (Undegassed) mmhos	1.0—2.0	1.0—2.0	1.0—2.0	2—10	1.0—2.0
Hydrazine ppm N_2H_4	—	—	—	0.01—0.05	—
Ammonia ppm NH_3	0.1—0.2	<0.1	—	—	—
Copper ppm Cu	<0.01	<0.01	<0.01	<0.01	<0.01
Iron ppm Fe	<0.01	<0.01	<0.01	<0.01	<0.01

are kept tight to eliminate leakage of cooling water into the system. Hydrazine is fed continuously into the deaerated feedwater at rates sufficient to maintain a measurable residual in the boiler water. Boiler blowdown is continuous at about 500 pounds per hour.

These operating practices result in the water conditions shown in Table 1. The iron and copper values reported were determined using batho-phenanthroline and neo-cuproine as colorimetric indicators in the respective test procedures; in the case of the iron determinations, the samples were acidified to ensure the solution of all iron present. It is felt that the accuracy of the tests is sufficient to readily establish that the actual concentrations are within the range shown and possibly substantially lower than the values shown.

Hydrazine concentrations were determined by the dimethylaminobenzaldehyde colorimetric procedure and ammonia concentrations by Nesslerization (ammonia is present as a reaction product of the hydrazine). The values or ranges of values shown represent the normal operating conditions observed in 13 boilers ranging in steam generating capacity from 620,000 pounds per hour to 1,000,000 pounds per hour. The boilers have been in service for periods ranging from 18 months to 9 years with five of the units being of the controlled circulation type.

Boiler Blowdown

Limits are observed on dissolved silica in the boiler water and these limits are normally the controlling factor in determining boiler blowdown rates. Silica is controlled at less than 3 ppm in the 1350 psig boilers and at less than 1 ppm in the 1850 psig units. A typical blowdown rate of 500 pounds per hour maintains these concentrations easily. Turbidity never is observed except for brief periods when the boilers are first started up.

The authors consider it desirable to maintain continuous boiler blowdown even where the required rate is low since this type of operation contributes to stable water conditions and ensures good boiler water sampling where the sample points are located in the blowdown line. However, during periods when plant operation has required strict conservation of water, some of these boilers have been operated for periods of several weeks without any blowdown except for the amount required to flush out the line for collecting the daily boiler water samples. No significant increases in the boiler water solids occurred during these periods.

Boiler internal surfaces are inspected annually and the surfaces are normally found to be covered with a light powdery coating which analyzes approximately 70 percent Fe_3O_4 and 25 percent copper with small amounts of nickel, zinc and other metals. Some of this material also is found lying in the drums of the boiler.

Tube failures have been experienced from oxygen corrosion in two of the boilers referred to and in each case it was found that oxygen was entering the boiler feedwater during periods of off-on or reduced load operation. Residuals of oxygen scavenging chemicals were lost during these periods. The tube failures were stopped by taking operating measures to minimize the introduction of oxygen into the boiler feedwater and by giving careful attention to the maintaining of oxygen scavenger residuals in the boiler water at all times.

All of the boilers were cleaned chemically for mill scale removal before being put into operation. It is the authors' intention to chemically clean for removal of oxide deposits when the visual inspection of tube samples indicates the need for removing the deposits. To date, only the two boilers which experienced corrosion failures have been chemically cleaned, one after eight and the other after seven years of operation. It is expected that several others will be cleaned in the near future and it now appears that these boilers probably will require cleaning at intervals of about five years.

None of the turbines receiving steam from these boilers ever have shown a measurable increase in stage pressures due to accumulation of deposits and none of the extraction heaters ever have required cleaning. The turbines are normally inspected after about three years of operation and there is a small amount of material found deposited on the blading at about the saturation point in the machine. This material is mainly silica and contains some magnetic iron oxide. The pH of the condensate is consistently about 8.5 and the turbine parts do not show evidence of any localized low pH conditions.

The operating experience described indicates to the authors that high pressure steam boilers can be operated without difficulty for extended periods with water containing relatively low solids content.

Aluminum Alloys for Handling High-Purity Water*

By W. W. BINGER[1] and C. M. MARSTILLER[2]

Abstract

Aluminum alloys have been used for more than 20 years for storage and distribution systems handling distilled water. Recently, it became possible to investigate experience with some 30 distilled water systems which had been in use for various periods of time up to 24 years. Many of these systems had been made entirely of aluminum. Several were composed entirely of tinned copper or brass and others were made up of a combination of materials including aluminum.

Results indicate that aluminum alloy storage tanks, tubing, fittings and valves are highly satisfactory for handling distilled water and do not cause significant contamination of the water being handled.

The increased use of the ion exchange method for purifying water has required equipment fabricated from materials in which such a water could be handled with a minimum of pick-up and discoloration. Since many processes require large quantities of a water of very low total solids content, it was natural to consider aluminum alloys for the fabrication of storage tanks and piping for handling demineralized water. 4.6.5

Introduction

IN THE PAST few years, industry has developed processes requiring large quantities of high-purity water. Nearly every chemical company, research or industrial laboratory, university or college, food manufacturer or pharmaceutical house requires high-purity water in some part of its operations. Aluminum alloys have been used for storage and distribution systems handling distilled water for more than 20 years because their use results in minimum contamination of the water.[1,2]

Distilled Water

Since distillation is the oldest of the water purification processes, it is natural that most of the experience gained over the past 20 years with aluminum alloys for handling highly purified water has been with the storage and distribution of distilled water.[3,4] Performance records have been accumulated on more than 30 distilled-water systems that have been in use for varying periods of time, some for over 20 years. Many of these systems were constructed entirely of aluminum alloys, some of tinned copper or brass, and some of combinations of these and other materials. The systems observed represent a wide range in geographic location and volume of water handled. In all cases, the systems are industrial installations handling relatively high-purity water.

The distilled-water systems described in Table 1 were constructed entirely of aluminum alloys. It can be seen from the data that the purity of the water contained in the systems remained high. In all cases where it was determined, the aluminum content was less than 0.02 part per million.

The systems described in Table 2 were constructed primarily of tin-lined copper or brass. Although the quality of water from these systems was, on the average, slightly lower than that from the aluminum systems, the pickup of tin and copper was low. Data obtained on a number of distilled-water systems, in whose construction a variety of materials was employed, both with and without aluminum, are presented in Table 3. The quality of the water from these systems is comparable with that found for the aluminum systems and the tin-lined copper systems.

In the collection of data for Tables 1, 2 and 3, emphasis was placed on the over-all quality of water from systems constructed of various materials. To supplement these data, 12 essentially all-aluminum systems shown in Table 4, representing different methods of operation and types of raw water supply, were examined for both total dissolved solids and aluminum content of the water. The average aluminum content of water from these 12 systems was less than 0.02 part per million. It was significant that there was no apparent correlation between age of the system and either the quality of the water or the aluminum content.

These data serve to demonstrate that the purity of the distilled water from the all-aluminum systems was generally very high and, on the basis of metal pickup, averaged somewhat lower than from the other systems evaluated. From the results obtained, it will be noted that the quantity of impurities, resulting from attack of the systems by distilled water, was very low. Probably the impurities in the water were more a function of still construction and operation than of the alloy composition of the storage tanks and distribution systems. In any event, the results indicate that aluminum storage tanks, distribution lines, fittings, and valves are highly satisfactory for handling distilled water because they result in minimal contamination of the water being handled. A typical all-aluminum system is shown in Figure 1. Additional factors that should not be overlooked include cost, formability, ease of installation, and availability.

Extensive alterations in one wing of a laboratory presented an opportunity to examine considerable aluminum alloy piping and fittings in the distilled-water system after 17 years of service. The 1100 alloy piping and cast 43 alloy fittings and valves were found to be in such excellent condition that all of this material was re-used in the new installation. In the extension of this distilled-water system, 3003

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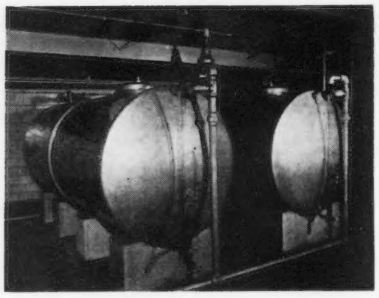

Figure 1—Typical all-aluminum installation for pharmaceutical plant requiring large quantities of distilled water. Storage tanks (2000 gallons) are of 3003 alloy and distribution system is of 3003 alloy pipe. Other features are standard cast B214 alloy fittings and cast 43 alloy valves.

alloy pipe was employed with cast B214 alloy fittings. Aluminum alloy 3003 pipe was selected because screwed joints were involved and this alloy has better threading characteristics than does 1100 alloy.

Effect of pH

If the quality of the distilled water is good (i.e., if water is low in total solids), there is substantially no corrosion of aluminum alloys, and the pH of the distilled water is of little concern. Laboratory tests have indicated that pH could have some effect on the resistance of 3003-H14 to corrosion by distilled water. Specimens (0.064 inch x 0.5 inch x 3 inches) of 3003-H14 were exposed for one week at 60 C in 250 ml of distilled water, the pH of which had been adjusted and maintained with either hydrochloric acid or sodium hydroxide to cover a pH range of 2.8 to 10. The results given in Figure 2 show that in the pH range of 4 to 9 the attack is low. Based on other data in this pH range, the attack tends to become self-limiting with increasing time of exposure. The results also show that the minimum weight loss occurred at a pH value of 4 to 4.5. This was rather surprising because normally the presence of chloride ion would be expected to increase, rather than decrease, the amount of corrosion. Although this effect is slight, it is believed that in certain cases the pH of the high-purity water employed should not be overlooked as a possible means of further improving the performance of aluminum alloys.

Effect of Temperature

The increasing use of aluminum alloys in the chemical industries has generated considerable interest in the resistance of aluminum alloys to corrosion by high temperature water and steam. Although aluminum alloys have been used satisfactorily for many years to handle steam and steam condensate at temperatures below 250 F, there was little or no information available concerning their behavior at higher temperatures.

The company's initial investigation on the behavior of aluminum alloys exposed to high-purity water at elevated temperatures was confined to alloy 3003-H14, one of the more common aluminum alloys used in the fabrication of chemical equipment. Specimens (0.064 inch x ½ inch x 3 inches) of alloy 3003-H14 were exposed to both distilled water and water vapor in stainless steel pressure vessels.

In all tests, one liter of distilled water was added to

TABLE 1—Analysis of Distilled Water From Systems With Aluminum Alloy Storage Tanks and Distribution Lines

Location	Description of System	Length of Service Years	Volume of Water Handled Gallons/ Week	Total Solids ppm	Aluminum Content ppm
Alabama	1100 alloy tank, tubing, and valves	9	300	0.2	....
Arkansas	Aluminum alloy tank, 1100 alloy tubing and rubber valves.	5½	400	0.1	....
Arizona	Aluminum alloy tank, 1100 alloy tubing, and cast aluminum alloy fittings.	5	100	0.6	0.015
California	3003 alloy tanks and tubing and tin-line brass valves.	7	200	1.1	....
Illinois	1050 alloy tank and 6061 tubing.	3½	200	0.4	0.001
Illinois	1100 alloy tank, 3003 alloy tubing and cast aluminum alloy fittings and valves	6	2500	0.4	....
Illinois	3003 alloy tank and tin-lined brass valve.	4	600	0.4	....
New Jersey	1100 alloy tank, 6061 alloy tubing and fittings and valves of 2017 alloy and cast 406 alloy.	11	300	0.1	....
New York	1100 alloy tank, 6061 alloy tubing, and 3003 and cast 43 alloy fittings.	10	400	0.5	....
North Carolina	1100 alloy tank and tubing and cast 406 alloy fittings.	22	100	0.2	0.003
Pennsylvania	1100 alloy tanks and tubing and cast 43 alloy fittings and valves.	19	400	0.4	0.004
Pennsylvania	3003 alloy tank and cast 43 alloy fittings and valves.	7	500	0.1	0.002
Tennessee	Aluminum alloy tank, no distribution system.	21	300	0.2	0.005
Washington	3003 alloy tanks and tubing, cast B214 alloy fittings, and cast 43 alloy valves.	9	600	0.1	....

TABLE 2—Analyses of Distilled Water From Tin-Lined Copper Systems

Location	Description of System	Length of Service Years	Volume of Water Handled Gallons/ Week	Total Solids, ppm	Metal Content, ppm	
					Copper	Tin
New Jersey	Tin-lined copper tank, no tubing and tin-lined brass valve	4	50	0.8	....	....
New York	Tin-lined copper tank, no tubing and tin-lined brass valve	9	100	4.4	0.4	0.001
Ohio	Tin-lined copper tank, no tubing and tin-lined brass valve	9	200	1.4	..	....
Ohio	Tin-lined copper tank, no tubing and tin-lined brass valve	5	50	0.1	..	....
Pennsylvania	Tin-lined copper tank, no tubing and tin-lined brass valve	5	50	0.2	0.01	0.001
Pennsylvania	Tin-lined copper tank, and tubing and tin-lined brass valve	5	50	2.3	..	....

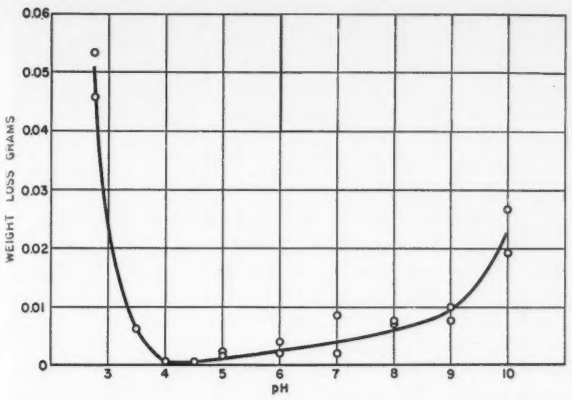

Figure 2—Effect of pH of distilled water on the weight loss of 3003-H14 alloy specimens (0.064 inch x 0.5 inch x 3 inches) exposed at 60 C for one week. Although the attack was low in the pH range 4 to 9 and tends to be self-limiting, note that the minimal attack occurs at pH 4 to 4.5 in spite of the added chloride ion.

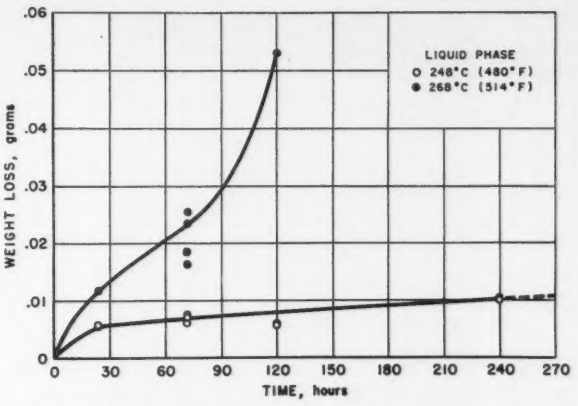

Figure 3—Effect of time and temperature on the weight loss of 3003-H14 alloy exposed to distilled water. For temperatures up to 248 C (480 F) the attack is self-limiting.

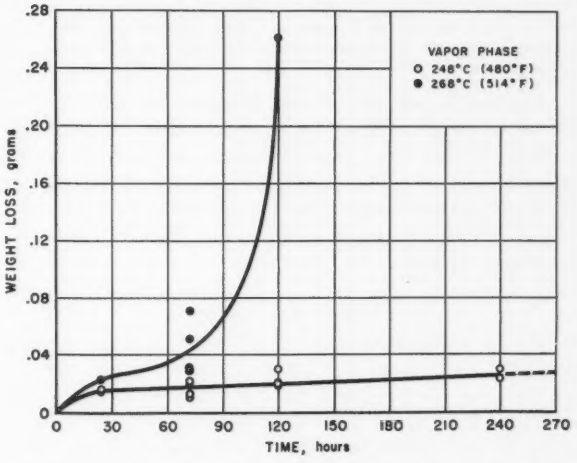

Figure 4—Effect of time and temperature on the weight loss of 3003-H14 alloy exposed to steam. The vapor phase was more aggressive than the liquid phase under identical conditions of temperature and pressure.

the test vessel having a capacity of two liters. For this investigation, temperatures up to 314 C (597 F) were investigated for exposure periods up to 240 hours. As a precaution against dissimilar metal contact between the specimens in the liquid phase and the stainless steel bomb, an aluminum oxide crucible was used to hold the specimens. Specimens exposed in the vapor phase were suspended on stainless steel wire hooks and insulated from the wire by means of quartz tube. Initially there was some question regarding the desirability of using alumina crucibles and quartz because of the possibility of contamination; however, subsequent tests using sapphire hooks to support and insulate the specimens indicated that contamination had not occurred.

It was found that 3003-H14 alloy exhibited good resistance to corrosion in both the liquid and vapor phase up to a temperature of 248 C (480 F). Data obtained in the liquid phase are shown in Figure 3. At temperatures up to 248 C (480 F) the attack tended to be self-limiting and the calculated rates of attack were less than 0.004 in.^{3}/in.^{2}/yr. When the temperature was increased to 268 C (514 F), the weight loss increased sharply after 72 hours of exposure. A further increase in temperature to 314 C (597 F) resulted in complete corrosion of the specimens in less than 24 hours.

The data shown in Figure 4 indicate that the behavior of 3003-H14 in the vapor phase was similar to that encountered in the liquid phase, but the vapor phase was slightly more aggressive. Up to a temperature of 248 C (480 F), the increase in attack was not significant for exposures of 120 hours duration; at 268 C (514 F), however, the rate of attack in the vapor

TABLE 3—Analysis of Distilled Water From Miscellaneous Systems

Location	Description of System	Length of Service Years	Volume of Water Handled Gallons/Week	Total Solids ppm	Aluminum Content ppm
Arkansas	Tin-lined copper tank, 1100 alloy tubing and cast 43 alloy fittings.	5	200	0.9	....
Arkansas	Pyrex storage carboy, glass tubing and rubber valve.	4	100	0.4	....
Connecticut	Tin-lined copper tank, 3003 alloy tubing and tin-lined brass valve.	6	100	1.1	....
Illinois	Tin-lined copper tank, 3003 alloy tubing and cast aluminum alloy fittings.	6	50	0.6	....
Michigan	Tin-lined copper tank, 3003 alloy tubing and tin-lined brass fittings and valves.	6	10	0.1*	0.001
New Jersey	Tin-lined copper tank, 1100 alloy tubing and cast 43 alloy fittings and valves.	5	100	0.4	....
New York	Tin-lined copper tank, 3003 alloy heating tank, 1100 alloy tubing and cast 43 alloy fittings.	10	100	3.0	0.05
Washington	Tin-lined copper tank and 1100 alloy tubing.	6	200	0.6	....
Washington	Tin-lined copper tank, aluminum alloy tubing, and tin-lined brass valves.	5½	100	0.6	0.002
Washington	Tin-lined copper tank, 6061 alloy tubing and 2024 alloy fittings.	5	200	1.3	....

* Copper content 0.098 ppm., tin content 0.00 ppm.

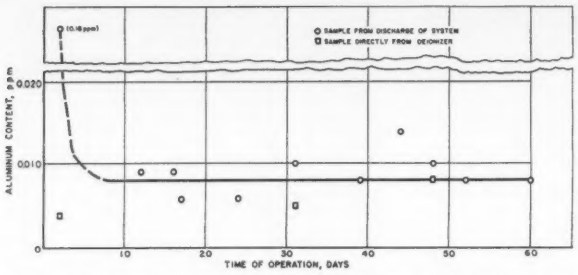

Figure 5—Aluminum content of deionized water handled in all-aluminum distributing system. Note that after the initial conditioning period the aluminum pick-up by the water was negligible.

phase was 0.125 $in.^3/in.^2/yr$ as compared to 0.025 $in.^3/in.^2/yr$ in the liquid phase.

On the basis of these data, 3003-H14 alloy probably should not be used to handle steam or high-purity water at temperatures over 232 C (450 F). Section 8 of the ASME Boiler and Pressure Vessel Code permits the use of 3003 alloy and certain other aluminum alloys at metal temperatures not exceeding 400 F.

Recently there has been considerable interest in the use of aluminum alloys at temperatures in excess of 450 F. An alloy development program is under way to develop alloys suitable for such service. Although considerable progress has been made in the development of such alloys, they are beyond the intended scope of this paper.

Deionized Water

Many requirements for pure water are now being met by the deionizing process rather than distillation. Since the problems of handling deionized water are much the same as for distilled water, it was natural to consider aluminum alloys for the fabrication of storage tanks and distribution systems to maintain the purity of deionized water.[5]

A new deionized water system presented an opportunity to follow the performance of a system and determine the aluminum pickup. This system, using a portable water softener and mixed bed deionizer, consisted of an all-aluminum distribution system, including a 6061 alloy surge tank, 350 feet of ⅝-inch diameter 3003 alloy tube, 6061 alloy compression fittings, and six cast 356 alloy valves. Water samples were taken periodically from the deionizer and from the discharge end of the distribution system. To check for the maximum aluminum pickup in the distributing system, water samples were taken in the morning before the system was started each day. Thus, it was possible to collect "stagnant" water samples that had been in contact with the aluminum alloys for 16 to 48 hours (system not used during week-ends). These data are tabulated in Table 5 and plotted in Figure 5. It is readily apparent that, during the initial period of operation as the system was being conditioned, the aluminum content of the deionized water increased appreciably. After the system was conditioned, however, the aluminum pickup by the deionized water was negligible.

Service Experience

Aluminum alloys have become standard materials of construction for handling deionized water. Several typical installations will demonstrate the type of performance to be expected.

Pharmaceutical Plants

Most pharmaceutical plants require large quantities of highly purified water in their manufacturing operations.[6] A typical storage and distribution system for deionized water is as follows:

Storage facilities include two 2,500-gallon and one 1,000-gallon tanks of 3003 alloy. The distribution system contains 800 to 900 feet of aluminum alloy pipe ranging in size from ½ inch to 1½ inch in diameter. The distribution system carries demineralized water from the deionizer to the storage tanks and from the storage tanks to the various operating divisions where the water is used for manufacturing and washing purposes. No maintenance except for an occasional cleaning of the tanks has been required in the six to eight years the system has been in operation.

Samples of deionized water are checked at weekly intervals in accordance with the analytical tests prescribed in the *U. S. Pharmacopoeia* for purified water. The total solids normally varied from 0.0020 to 0.0035 gram/liter (2 to 3.5 ppm). An analysis of a recent water sample revealed that the aluminum content was only 0.024 ppm.

Steam Power Plants

Modern, high-pressure steam power plants require large quantities of high-purity water for steam generation. In such plants, how-

TABLE 4—Analyses of Distilled Water From Typical Aluminum Alloy Storage and Distribution Systems

LOCATION	DESCRIPTION OF SYSTEM				Length of Service Years	Volume of Water Handled Gallons/Week	Total Solids, ppm	Aluminum Content, ppm
	Tanks	Lines	Fittings	Valves				
Alabama	1100	1100	B214	B214	15	350	0.45	0.019
Arkansas[1]	3003	3003	B214	B214	3	1700	0.22	0.092
California	3003	3003	406 43	B214	13	5	0.42	0.004
Illinois	1100	3003	Tin-lined brass	Tin-lined brass	12	2500	0.78	0.030
Illinois	1100 3003	3003	43	43	10	600	0.16	0.002
Indiana	3003	3003	5053	B214	15	80	0.95	0.010
New Jersey	1100	6051	406 2017	43	17	200	0.52	0.000
Pennsylvania	1100	1100	43	43	26	500	0.61	0.013
Tennessee	1100	....	....	Tin-lined brass	10	300	5.47	0.038
Texas	3003	3003	43	43	3	300	0.37	0.004
Washington	3003	3003	406 B214	B214	14	300	0.17	0.001
Washington	3003	3003	43 B214	43 B214	3	200	0.07	0.001

[1] Alumina refining plant. High aluminum content attributed to alumina dust in atmosphere.

ever, the use of aluminum alloys for handling deionized water is a relatively new application. Approximately one year ago, an aluminum alloy distribution system was installed by an eastern power plant to handle deionized make-up water for two boilers. In this case, the storage tank was wood. The distribution system contained 400 feet of 2½-inch and 3-inch diameter aluminum alloy pipe and aluminum alloy valves and fittings. After approximately one year of operation, an analysis of the water revealed that the total solids were 1.41 ppm and the aluminum content was only 0.014 ppm—1 percent of the total.

Chemical Plant

In one chemical plant, the deionized water system in service 3½ years has a 3,000-gallon storage tank of 3003 alloy and a distribution system of Type 304 stainless steel. The total solids in the water in the storage tank were 3.53 ppm and the aluminum content was 0.019 ppm.

Another deionized water system at the same plant has a 3,000-gallon storage tank of Type 304 stainless steel, 200 feet of 1100 aluminum alloy pipe (1½-inch and 2-inch diameter), aluminum alloy fittings, and Type 304 stainless steel valves. It was reported that the quality of water from both systems was entirely satisfactory.

In another chemical plant, the deionized water systems, consisting of a 6061 aluminum alloy storage tank and a plastic pipe distribution system has been in service for eight years. It was reported that the aluminum pickup of the water was nil.

Based on economy and the maintaining of quality of deionized water, it is believed that an all-aluminum system is justified.

Atomic Energy Applications

Probably the most publicized use of aluminum for handling high-purity water is for atomic energy applications.[7,8,9] In addition to cost factors, the selection of materials depends on availability, good nuclear properties, resistance to corrosion, heat transfer, tensile strength and creep resistance at high temperature, formability and ease of welding, resistance to radiation damage and dimensional changes from irradiation and resistance to radioactivity build-up in the material itself. It has been reported[10] that aluminum alloys appear to be one of the most promising materials of construction because they combine many of these essential properties.

Steam Condensate

Corrosion by steam and steam condensate in systems other than aluminum has been a problem frequently encountered in many plants. A principal cause of corrosion by steam and steam condensate is dissolved oxygen and carbon dioxide. Aluminum alloys are particularly well suited for handling steam or steam condensate[11] containing carbon dioxide or oxygen because these contaminants have no adverse effect on aluminum alloys. In fact, aluminum alloys have been used for handling steam and steam condensate for over twenty years in the chemical and food industries.

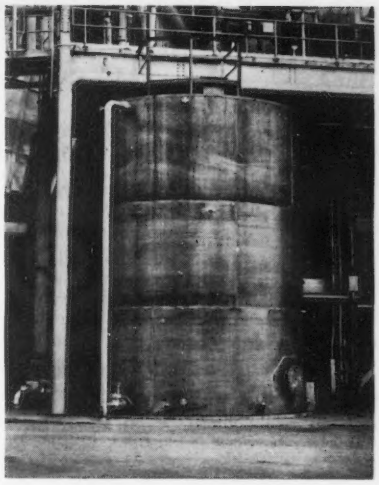

Figure 6—One of the three 18 foot diameter, 30 foot high aluminum condensate storage tanks. Aluminum alloys are particularly well suited for handling steam condensate containing carbon dioxide or oxygen because these contaminants have no adverse effect on aluminum alloys.

TABLE 5—Determination of Aluminum Pick-Up by Deionized Water Handled in an Aluminum Alloy Distribution System

Time From Start-Up Days	Water Sample	Total Solids,[1] ppm	Aluminum, ppm
2	Directly from deionizer	0.51	0.004
2	Held in system 48 hours[2]	1.75	0.18
12	Held in system 16 hours[3]	0.81	0.009
16	Held in system 48 hours	0.91	0.009
17	Held in system 16 hours	0.81	0.006
24	Held in system 16 hours	0.62	0.006
31[4]	Directly from deionizer	5.06	0.005
31[4]	Held in system 16 hours	4.54	0.010
39	Held in system 16 hours	0.61	0.008
44	Held in system 16 hours	0.41	0.014
48	Directly from deionizer	1.21	0.008
48	Held in system 16 hours	0.89	0.010
52	Held in system 16 hours	0.74	0.008
60	Held in system 16 hours	2.08	0.008

[1] Total solids at 105 C.
[2] Water sample taken from the system after being idle over the week-end.
[3] Water sample taken from system in morning before start-up.
[4] Deionizer regenerated after this sample.

The entrainment of alkaline boiler compounds can present a hazard to the performance of aluminum alloys. It is common practice to "trap out" alkaline boiler compounds upstream from aluminum piping. In many systems, amines or ammonia compounds are used to reduce the corrosive nature of steam and steam condensate. Although it is not necessary to add these compounds to prevent corrosion of aluminum, experience has indicated that in the concentrations normally employed, these materials have virtually no effect on the performance of aluminum alloys.

The use of aluminum alloys for storage of steam condensate in power plants represents a relatively new and excellent application. High-quality steam is needed to prevent attack of the turbines, particularly in modern, high-pressure power plants. In some

power plants, silicate deposits (carry-over from the boiler) tend to form on the turbine blades necessitating periodic cleaning. Initially there was some concern regarding the use of aluminum alloys for storing condensate or deionized water because it was believed that if aluminum silicates formed they would be extremely difficult to remove from boiler tubes and turbine blades. Field tests by one utility allayed their concern and has resulted in the use of 3003 alloy tanks to alleviate their condensate storage problem. An analysis of the water after three months of service in the system showed an aluminum content of only 0.04 ppm.

The tank shown in Figure 6 is one of three aluminum storage tanks 18 feet in diameter and 30 feet high in use for two years in condensate service to preserve condensate quality.

Service Considerations

The information from this survey has demonstrated that distilled or deionized water handled in aluminum alloy equipment was maintained at a high degree of purity. Although many aluminum alloys can be used satisfactorily to maintain the quality of purified water, the following alloys are normally recommended:

Tanks	— 1100, 3003, 5052 or 6061
Tube or Pipe	— 1100, 3003, 5050, 6061 or 6063
Cast Fittings	— 43, B214 or 356
Valves	— 43, B214 or 356

Before an aluminum storage and distribution system is put into operation, it is desirable to condition the system to build up an insoluble and protective film of hydrated aluminum oxide.[12] The following conditioning cycle has been employed successfully:

1. Flush with tap water to remove dirt, chips, etc.
2. Fill with tap water and allow system to stand 24 hours.
3. Flush, refill with tap water, and allow system to stand 24 hours.
4. Drain, flush system with high-purity water, fill, and let stand 24 hours.
5. Drain and refill. The system is ready for service.

Occasionally there have been reports of "cloudy" water, but generally the cause of this condition has been traced to entrainment or carry-over of impurities from the deionizing or distilling equipment.

Conclusions

It has been demonstrated that aluminum alloys are being used successfully for the storage and distribution of both distilled and deionized water. Although the maintaining of quality or purity of water is of prime importance, other factors that should be considered include low cost, availability, formability, and ease of handling. A sound, practical installation can best be obtained by taking advantage of all the information and experience that is readily available.

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Any discussions of this article not published above will appear in the December, 1957 issue.

The Formation of Oxide Films On Chromium and 18 Cr-8 Ni Steels*

By H. J. YEARIAN,* W. D. DERBYSHIRE* and J. F. RADAVICH*

Abstract

The formation of oxide films on simple chromium steels and 18-8 stainless steels oxidized in air at temperatures of 300 C to 700 C has been studied by electron microscopy, electron diffraction and X-ray diffraction methods. A thin uniform film forms in the first few minutes of oxidation and grows very slowly thereafter. As oxidation proceeds the protectiveness of the base film fails at randomly distributed positions, permitting the rapid growth of localized polycrystalline nodules of oxide. These nodules continue to grow and new ones are formed until the base film is completely covered. The rate at which the process proceeds depends on alloy composition and temperature in the same way as does the scaling rate at higher temperatures.

There is no evidence that the failure is due to cracking of the base film. There seems to be no detrimental effect produced by the usual amounts of minor constituents in the alloys. Local failure may be due to blistering or recrystallization.

The principal component of the base film is a solid solution of ferric and chromic oxides preferentially oriented with the (111) plane parallel to the surface. The degree of the orientation and the chromic oxide content of the solution increase with time of oxidation. A small amount of spinel may be present also. In the case of the 18Cr-8Ni alloy the spinel contains some Ni. The nodules are principally αFe_2O_3 or a low chrome ferric-chromic oxide solution and contain somewhat more spinel than does the base film. **3.2.3**

Introduction

DETAILED X-RAY diffraction investigations of the oxide scales formed on chromium and nickel-chromium steels oxidized in air or oxygen have been described recently.[1] The complementary studies of the structure and growth of the thin oxide films formed on these steels at lower temperatures or in shorter times than are necessary for true scale formation, have been made by electron microscope and by electron and X-ray diffraction methods. A brief report of part of this work was given some time ago[2]; similar investigations of complex heat resistant alloys have been described by one of the authors (J. F. R.).[3]

Methods and Procedures

The alloys investigated were in the form of wrought sheet and had the compositions shown in Table 1. These included a group of conventional chromium steels, a group of 26 percent Cr-steels with variable minor constituents and 18Cr-8Ni stainless alloys of the 303 and 302 types. A group of miscellaneous alloys was included for test of the generality of some of the conclusions. The work reported here is confined to air oxidation for various times at temperatures of 300 C to 700 C. Less extensive investigations show that oxidation in pure oxygen gives similar results.

The condition of the alloy surface has considerable influence on the way the metal will oxidize. Since any form of mechanical polishing leaves the surface in a disturbed state, anodic electrolytic polishing was used in all the work that follows, except as otherwise indicated. The electrolyte used was 2 parts 70 percent perchloric acid, 7 parts ethanol, 1 part glycerine.

Electropolishing conditions were adjusted to prevent etching. The polished sample was double rinsed in methanol and degreased. No differences in the oxidation were observed when ethylene dichloride, acetone or ethanol-ether was used as a vapor or liquid degreasing agent.

Several methods have been used to strip oxide films from the base metal to permit observation by transmission electron diffraction and microscopy. By scribing through the film to expose the metal, the action of an agency which dissolves the metal will loosen the film. This may be done chemically with bromine in methanol solution[4] or electrolytically in either KCl solution[5] or in the polishing electrolyte described above.[6] The authors prefer the latter method since the time of removal is only 2 to 10 seconds. Mechanical stripping with a heavy Formvar layer which could subsequently be dissolved away was not found to be generally applicable, but it was possible to show by this means that no observable change in the structure or composition of the oxide film was produced by the chemical or electrolytic stripping.

The oxidation of alloys is a complex problem which requires the full exploitation of as many pieces of experimental information as possible. Electron microscopy (RCA Model EMU-2) utilizing the chromium showcast Formvar replica technique gave information concerning the physical conformation of the outer surface of the oxide. The effect of successive oxidation exposures could be followed. When the film was stripped from the metal, those parts of it which were thinner than approximately 1000Å

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could be examined in transmission; the resolution was approximately 50Å. The disadvantage of this technique was that the oxidized surface was lost to further treatment.

Electron Diffraction

The principal method which was used to identify the oxides present was electron diffraction in its two forms. The exposed surface of the oxide was examined by diffraction at grazing incidence ("reflection" method, EDR). Interpretation of these patterns was subject to the usual severe limitations of this technique. The diffraction lines were usually broad; the true center of the pattern and the effective distance from sample to photographic plate frequently were ill defined. Because of these factors identification was not complete. While it was possible to distinguish the spinel type oxides, Fe_3O_4, $FeCr_2O_4$ and their solid solutions from rhombohedral phases, αFe_2O_3, Cr_2O_3 and their solutions, only rarely could one differentiate members within each phase type. These phases will be indicated simply as S and R, respectively, in reflection data. A small amount of the spinel could not be detected, when in the presence of the rhombohedral phase, since all of the stronger lines of the spinels are in close coincidence with rhombohedral lines. Preferred orientation was determined from the arcing of the diffraction rings.

Several important factors had to be considered in interpreting the reflection data. Because of the small grazing angle of incidence, combined with the limited path which the electrons could traverse without excessive inelastic scattering, the depth of penetration into a flat film surface was only 10 to 20 Å. No information was obtained about the sample below that depth and the lines were poorly resolved. To obtain a really useful pattern the surface had to contain a reasonable number of projections at least 100 Å high and not more than about 1000 Å wide in the beam direction through which the electrons could penetrate. If they were wider than this the pattern represented only the surface of the projections. There was the additional question of what fraction of the smaller projections or of the flat surface was eclipsed by larger projections. Full and proper use of the reflection diffraction data required a detailed knowledge of the mechanical character of the surface examined, so that a combination of electron diffraction and replica electron microscopy was called for.

Transmission electron diffraction (EDT) was applied to stripped films with greater confidence than was possible in reflection, and gave much better resolution of phases, determination of lattice parameters, composition of solid solutions, etc. Here also, monitoring of the specimen by use of transmission electron microscopy was essential since only those parts of the film which appeared transparent in the micrograph could contribute appreciably to the diffraction pattern. Preferred orientation with a fiber axis perpendicular to the film was determined by the arcing of the diffraction rings when the film was inclined to the beam.

X-ray diffraction is not usually considered to be applicable to the study of thin oxide films. Sometimes it was possible, however, to wrap several small pieces of stripped film around a fine Pyrex fiber in such a way that a usable pattern could be obtained in a small Debye-Scherrer camera with a ten to fifteen hour exposure. Although these patterns were not as strong as conventional ones, they usually gave better resolution of phases and composition of solid solutions than did the corresponding EDT patterns. More important than this advantage, however, was the fact that the relative intensities of the patterns of the various phases were a measure of the relative amounts of those phases present in the whole sample, whereas EDT patterns pertained to the parts thinner than about 1000 Å and EDR patterns to the surface.

TABLE 1—Composition of Alloys (Weight Percent)[1]

SAMPLE	Cr	Ni	C	Mn	Si	P	S	N
AM-5	5.76	0.095	0.051	0.48	0.44	0.008	0.006	.....
AM-13	13.72	0.26	0.077	0.40	0.23	0.018	0.029	.....
AM-17	17.45	0.33	0.072	0.46	0.59	0.018	0.017	.....
AM-26	26.31	0.33	0.14	0.68	0.74	0.020	0.013	0.115
K-19	18.95	.....	0.01	.....	.....	.....	.....	0.002
V-1	25.20	.....	0.016	0.008	0.14	.....	.....	0.010
V-2	26.01	.....	0.022	0.12	0.16	.....	.....	0.016
V-3	26.20	.....	0.024	0.55	0.15	.....	.....	0.018
V-4	25.32	.....	0.027	0.69	0.50	.....	.....	0.043
V-5	26.31	0.33	0.14	0.68	0.74	0.020	0.013	0.115
302	17.0 —19.0	8.0 —10.0	0.080 —0.20	2.00 max.	1.00 max.	0.040 max.	0.030 max.	....
303	18.37	9.16	0.13	0.63	0.37	0.021	0.012	.....
W-4	18.96	80.80	0.03	0.0	0.01	.....	.....	.....
446	23.0 —27.0	.....	0.35 max.	1.50 max.	1.00 max.	0.040 max.	.....	.....
Die Steel	0.25	.....	0.60	0.25	.....	.....	.....	.....
1020	.....	.....	0.18 —0.23	0.30 —0.60	.....	0.04 max.	0.05 max.	.....

[1] Balance Fe.

Physical Structure and Growth of Oxide Films

In the first systematic EDT investigations which were made of the films produced on the AM series of chromium steels, little difference in their composition was observed. They were found to consist of a rhombohedral phase containing more or less Cr_2O_3 in solution with αFe_2O_3 as the primary component and a small amount of a spinel. The electron micrographic examination of these films, however, revealed very striking differences in the physical nature of the films. Since an understanding of these differences is essential to an interpretation of the diffraction data, the micrographic studies will be considered first.

When one of these alloys is heated in oxygen or air for a short time to a temperature of 500 C to 700 C (e.g., AM-17 for 1 minute to a maximum temperature of 650 C) a thin, quite uniform temper-color film is produced which when stripped from the metal has the appearance shown in Figure 1a. The film is composed of a uniform thin mat of crystals whose size is in the range of 100-300 Å. The mean thickness of the film is of this same order and usually does not vary by more than a factor of about two from one metal grain to another. A few larger crystals approaching 1000 Å in size, and therefore nearly opaque

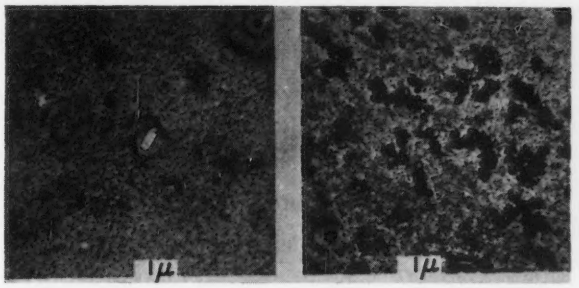

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Figure 1—One minute cycles of heating AM-17 in air to a maximum temperature of 650 C; (a) 1 cycle, (b) 25 cycles. Positive print electronmicrograph of stripped oxide film; opaque regions black.

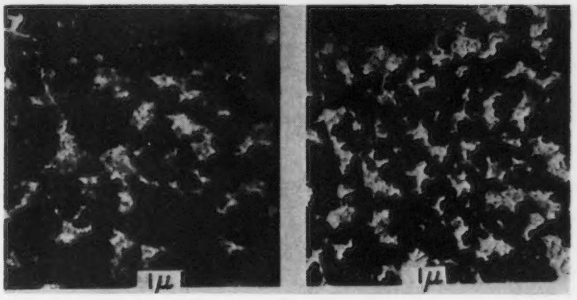

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Figure 2—AM-13 and AM-26 oxidized 20 hours in air at 600 C; (a) AM-13, (b) AM-26. Positive print electronmicrograph of stripped oxide film; opaque regions black.

to the 50KV electrons, may also be visible (the small black specks). The white spot near the center of the photograph marks the position of a small carbide inclusion in the metal surface over which an exceedingly thin and structureless film has grown. Such carbides usually protrude slightly above the general surface, due to their slower attack during the polishing process.

In addition to this thin film, which will often be referred to as the base film, a few opaque regions larger than the small black specks may be visible, as in Figure 1a. On increasing the oxidation time (e.g., to 25 heating cycles as shown in Figure 1b), one finds three changes have occurred. The thickness and the crystal size of the base film have increased by a factor of perhaps two in the case illustrated, but the number of the larger opaque regions has increased several-fold. With further oxidation this process continues in the same general way until the whole film becomes covered with the irregularly shaped opaque regions, the base film itself having increased in thickness by a relatively small amount.

What has been illustrated with AM-17 heated in one minute cycles to 650 C is in no way limited to these conditions. Any of the chromium and 18Cr-8Ni steels exhibit a similar behavior for both cyclic and continuous oxidation. The time scale is different, however, for the different alloys and for different temperatures. The process is accelerated by a decrease in chromium content of the alloy and by an increase in temperature of oxidation (i.e., the dependence is analogous to the dependence of scaling rate on these variables at higher temperatures). Figure 2 shows that after 20 hours of continuous oxidation at 600 C the base film formed on AM-13 is nearly covered with the localized oxide growths while AM-26 is about half covered. Under the same conditions the nodular oxide on AM-5 coalesced to a continuous optically opaque layer from which the electron-optically transparent base film separated during the stripping process. Microphotographs of 5 percent and 13 percent Cr-Fe given by Gulbransen and co-workers[7,8] unquestionably show similar effects of local attack as do photographs of other samples which are described simply as "non-uniform" films.

Opaque Crystalline Masses

In the growth illustrated in Figure 1, an appreciable fraction of the metal oxidized during the interval must have gone to form the opaque crystalline masses, and at an advanced stage (Figure 2), their growth involves the majority of the total metal loss. It is felt therefore that this phenomenon is an important part of the thin film oxidation process, and that disregard of the large masses has introduced numerous misinterpretations of experimental data. There is only one theoretical treatment[9] known to the authors which does not assume a uniform film.

When the exposed surface of the oxide film is investigated by the replica method it is found that nodules of oxide appear on the film surface. Several of these at early stages of formation are visible in Figure 3. Large numbers of such replica micrographs of the surface have been compared with the transmission photographs of the same films; in no case were the surface protuberances insufficient, either in shape or number, to account for the opaque regions observed in transmission. It is clear, therefore, that the inside surface of the base film is roughened during oxidation by a comparatively small amount.

Use of Landmarks

In order to follow the process of local nodular oxide growth in more detail, a technique was developed whereby the same field of the replicated surface could be relocated after various treatments of the sample, using natural landmarks such as carbide inclusions or artificial ones produced with a microhardness indenter.

Figures 3a and 3b illustrate two early stages of oxidation of AM-26. The large smooth surface at the lower right and smaller ones at the upper right and lower left of the photographs are carbides standing in relief. Stripped films of control specimens prove that a thin film similar to that in Figure 1a covers the matrix material and is very thin over the carbides. There is a faint trace of a grain boundary extending from the tip of the large carbide upward to the left, so that parts of two grains of the matrix are in the field. In photograph 3a it is evident that the right grain has started to develop more and larger nodules than has the left. After a longer oxi-

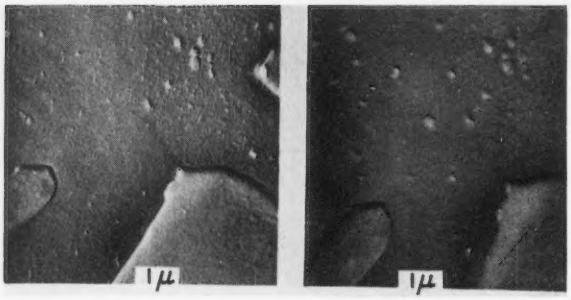

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Figure 3—Oxidation of AM-26 in air at 500 C; (a) 34 minutes, (b) 94 minutes. Positive print electronmicrograph of shadowcast formvar negative replica of oxide surface. Elevations of surface appear light, shadows white.

TABLE 2—Summary of Electron Micrographic Investigations—Air Oxidation

ALLOY	(Pre-treatment, other than Electro-Polish.) Oxidation Conditions.	Examination[1]
5 to 10 Samples Each of:	Successive One Minute Cycles to Max. Temperature of 650 C	
AM-5	0 to 13 cycles	
AM-13	0 to 19 cycles	R, T, after first and every 3 to 10 cycles.
AM-17	0 to 25 cycles	
AM-26	0 to 57 cycles	
AM-26	(etched) 0 to 33 cycles	R
All AM-(4 samples)	0, 5, 30, 1080 minutes at 500 C. Repeat at 575 C.	R, T
All AM-	0, 30 min. at 625 C.	R, T
All AM-	0, 20 hours at 600 C.	R, T
All AM-(3 samples)	0, 30, 960, 1200 minutes at 625 C.	R, T
All AM-(2 samples)	0, 30, 60 minutes at 700 C.	R, T
All AM-	0, 4, 14, 34, 64, 94, 140 minutes at 500 C. Repeat at 550 C.	10 fields RR followed by SR
K-19;AM-17,26; AM-17*,26*	(AM-17*,26*, H_2 annealed) 0, 5, 15, 45 minutes at 550 C.	5 fields RR followed by SR
V-1,2,3,4,5 (5 samples)	0, 15, 45, 90, 600 minutes at 600 C.	T, R, followed by SR
(15 samples) AM-26	(Variation of electropolish and degreasing conditions) Various times at 600 C—650 C.	R
All AM-,446,1020, Die Steel,303,W-4 (3 samples)	0, 45, 90, 810 minutes at 300 C.	R, T
All AM-,446,303 (2 samples)	0, 15, 30 minutes at 500 C.	R, T
AM-26	(Short, medium, long electropolish; 3/0 paper polish, diamond polish.) 16 hours at 625 C.	R, T
303 (8 samples)	0, 5, 10, 15, 45, 105, 705, 2145 minutes at 550 C.	R, T, SR
303 (7 samples)	0, 5, 10, 15, 45, 105, 705 minutes at 650 C.	R, T, SR
303 (4 samples)	0, 5, 10, 15 minutes at 700 C.	R, T, SR
302	(Carbides in equilibrium) 0, 5, 15, 45, 105, 705 minutes at 550 C.	10 fields RR

[1] **Examination:** By replica, R; by transmission, T; by replica after stripping with Scotch tape, SR; by replica of relocated field, RR.

dation time, Figure 3b, new nodules have formed on both grains and the older ones have grown slightly.

From many studies of this kind, several conclusions are drawn:

1. The degree of the attack and the length of the induction period before nodular growth begins may vary by factors of two or three on differently oriented metal grains.

2. During the induction period the nucleus from which the nodule will grow is often visible as a small (e.g., 500 Å) projection which does not change during the quiescent period. Nodules, however, may also start to grow without previous indication of any irregularity in the film.

3. Initially a nodule is composed of polycrystals of 200 to 500 Å particle size but grain growth occurs as oxidation continues; the crystal size may become as large as 0.5μ by the time coalescence of the nodules has occurred. (These observations are confirmed by the diffraction experiments.)

4. Comparison of the effects produced by one minute heating cycles with continuous heating at a corresponding temperature gives no evidence that the local growths are induced by thermal stresses.

5. There is no indication of a crack developing in the film as nodular growth begins, although it is true that the rapid growth sometimes takes the form of connecting veins and ridges or irregular strings of rounded nodules. Such networks show no systematic directional effects.

6. Assuming the rounded type of nodule is approximately hemispherical in shape, measurements indicate that the volume of the nodule is roughly proportional to the logarithm of the time after the beginning of its growth.

The foregoing description of the growth of thin oxide films and the local failure of their protective qualities has been given in terms of observations on chromium steels. Many experiments of a similar nature have been carried out with several other types, particularly the Ni-Cr steels. A summary of these micrographic investigations is given in Table 2. Most of these experiments included diffraction experiments as described in the following section.

In these investigations involving several different types of steel under many different conditions of exposure, the general course of events was always the same. A thin uniform film was formed quickly. At random times during the subsequent slow growth of this film its protectiveness failed at isolated randomly distributed points, and a rapid local oxidation occurred in the form of nodules. This process continued until an incipient scale covered the whole film. The essential difference in behavior of the various alloys was that the time or temperature required to reach a given stage of development varied in the same general way as does their resistance to scaling at higher temperatures. More complex heat resistant alloys show similar effects.[3]

Stripping Off of Oxide Film

After an initial oxidation period it is sometimes possible to strip off parts of the oxide film with Scotch tape. In this event some interesting features of the attack are revealed, as shown in Figure 4. Photograph 4a is of a normal film before nodular growth has proceeded extensively; a grain boundary runs upward and downward to the left from the tips of the central carbide. Photograph 4b, taken after the mechanical stripping, shows that the film has

been removed at the top and at the right. Around the carbide and to the lower left, however, some oxide remains. It is evident that a top layer of oxide has been removed from the latter region, showing that the base film, at least after long times of formation, probably consists of two layers. The outer layer is perhaps two or three times as thick as the inner. Effects similar to this have been observed many times and corroborative evidence of the two layers is apparent in micrographs of stripped films.

The two metal grains rendered visible by the stripping procedure evince quite different degrees of attack during the oxidation. The grain at the right shows only a slight "lemon peel" effect while the one at the top has developed deep pits. Also, a deep trench has formed along the grain boundary. It is obvious for the deeply pitted grain (and superposition of the plates will verify it for the other grain), that there is no correlation between the positions of the surface nodules and the pits in the metal. This is also the case at more advanced stages of oxidation where the nodules are more pronounced.

Note that there is no evidence on the surface of the oxide film of the presence of the deep pits and trenches below the film. Under the most severe conditions of attack, (e.g., 100 hours at 625 C), the pits may become a micron or more in diameter without external evidence of their presence; they often develop crystallographically oriented bounding surfaces.

Pits Empty of Oxide

Two pieces of indirect evidence lead to the tentative conclusion that the pits are empty of oxide. No debris has ever been found in the pits so any oxide present must have come off with the oxide film. In several instances pieces of film have pulled off with a replica of the oxide film and these have not shown the large opaque regions which would correspond to the oxide in a pit. To convert the metal to oxide within the same volume would require a compression of the oxide to approximately 50 percent of its normal value. It is difficult to believe, therefore, that the compressional forces arising from the formation of an oxide within the confines of the metal would not bulge or crack the overlying film.

Assumption that the pits are empty of oxide poses the problem of how the metal is transported from the pits to all parts of the film in such a uniform manner as the observations require. Film and nodular growth goes on as rapidly over the pits as elsewhere, requiring transport over lateral distances which are too large compared with the film thickness to be accounted for by bulk diffusion.

The alternatives are transport by vaporization or by surface and intergranular diffusion. Vaporization has been considered as effective in producing voids in diffusion couples[10] but this seems to be unlikely at the temperatures concerned. The second alternative is more probable since these processes are known to increase in importance, as compared with volume diffusion, at low temperatures.

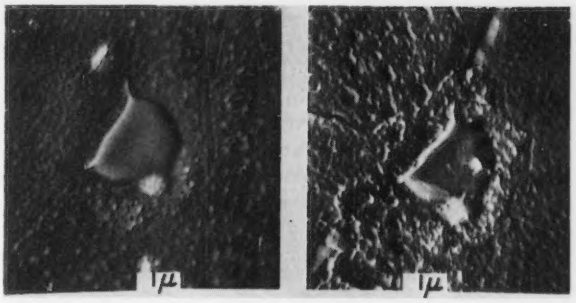

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Figure 4—Oxidation of AM-26 in air at 550 C for 140 minutes; (a) surface of oxide, (b) same after partial stripping with Scotch tape. Positive print electronmicrograph of shadowcast formvar negative replica; depressions in surface appear dark, shadows white.

Some additional general remarks can be made. Although the local attack was described above as occurring at random positions, a degree of regularity occasionally is observed. The growths may agglomerate into clusters or irregular chains. In some cases a vein-like formation of low narrow ridges may be produced before more isolated growths occur. These ridges rarely continue to grow excessively. It occasionally happens that the rounded, more or less isolated granules appear to be more transparent at the center than at the periphery when viewed in transmission, but by replica they show no sign of a crater in the external surface. This type of nodule must have a hollow dome shaped structure. This form seems to occur most often on chromium steels of high chromium content and on 18-8 stainless steels.

The number and size of the local growths usually varies somewhat from grain to grain of the alloy. Often there is a tendency for the local attack to increase in the vicinity of carbide inclusions and grain boundaries, although the reverse trend also has been observed. These inhomogeneous distributions may be associated with a gradient of a protective constituent of the alloy (e.g., chromium), or with an inverse gradient of a deleterious element. For example, dissolved carbon might be expected to weaken the film by reduction reactions. In the case of the chromium and nickel-chromium steels, growth on the carbides themselves occurs very rarely; as the surface density of the nodules increases they gradually encroach upon the thin film over a carbide and eventually cover it. Nodules do tend to form on the carbide inclusions of the iron-rich steels.

Discussion of Micrographic Observations

There are several possible causes of breakdown of the base film to be considered. The simplest of these are mechanical rupture, cracking, blistering, etc. These all involve some form of stress in the base film.

Thermal stresses do not seem to be the primary cause since the temperature cycled tests give no obvious increase in the attack. The failure does not give the impression of crack formation; it is difficult to see how rupture at what is virtually a point could relieve much stress. Blistering due to loss of adhe-

sion would seem to be more likely, particularly in view of the occasional formation of hollow nodules.

Pertinent to all explanations depending upon stress is the observation made in the following section that the films show a preferred orientation of the fiber type with random orientation about the film normal. They are not oriented pseudomorphic overgrowths and therefore the large stresses envisaged by Evans[11] should not be involved, as has been shown by Frank and van der Merwe[12] and discussed by Cabrera and Mott.[13]

Recrystallization

Local recrystallization of the film might be expected to lead to an increased attack since any of the protective qualities which are effective could be altered. Recrystallization seems not unlikely to occur because the thin film is formed very rapidly at far from equilibrium conditions, and as the film growth slows down quite other phases might be more stable. In the present case, however, the crystals in the nodules are isomorphic with those in the film although they differ somewhat in composition, as will be shown in the next section. Strong changes in protective qualities dependent on bulk properties such as diffusion coefficient do not seem likely to occur, but in the reorganization process intergranular diffusion might become significantly larger.

In addition to diffusion coefficients (or ionic transport numbers) and electrical conductivity which are the controlling factors in a Wagner[14] type theory of film growth, other factors may be effective at the interfaces to control the overall growth.[9,13,15] These factors are: 1. The activation energy for solution of a metal ion in the oxide lattice. 2. The energy to move an electron from the metal to the conduction band of the oxide. 3. The adsorption energy of oxygen on the oxide surface. 4. The work function of

TABLE 3—Composition of Oxide Films on Chromium Steels

Time and Temp. of Oxidation	Method	AM-5			AM-13			AM-17			AM-26		
5 minutes 500 C	EDT	—		R5 aaa	—		R10 aaa	—		R10 aaa	—		R5 aaa
	EDR	vw S a	(N)	R aa	w S bbb	(N)	R aaa	vw S ?	(N)	R aaa	—	(F)	R aaa
30 minutes 500 C	EDT	—		R5 aaa	—		RO aaa	—		R15 aaa	—		R22 aaa
	EDR	vw S a	(N)	R aa	w S bbb	(N)	R aaa	w S bbb	(N)	R aaa	vvw S ?	(N)	R aaa
	EDR inner	—		R	—		R	—		R	—		R
18 hours 500 C	EDT	—		RO aaa	—		R10 aaa	—		R20 aaa	—		R40 aaa
	EDR	vw S a	(T)	R a	vw S ?	(T)	R a	w S a	(N)	R a	—	(N)	R aa
5 minutes 575 C	EDT	vw LPS ?		RO aaa	—		RO aaa	—		RO aaa	—		R15 aaa
	EDR	vw S a	(N)	R aa	vw S ?	(N)	R aaa	vw S ?	(N)	R aaa	vw S ?	(N)	R aaa
30 minutes 575 C	EDT	—		RO aaa	—		R5 aaa	—		R30 aaa	—		R50 aaa
	EDR	vw S ?	(N,T)	R	—	(T)	R a	—	(T)	R aa	vw S ?		R a
18 hours 575 C	EDT				—		R40 aaa	—		R40 aaa	—		R65 aaa
	EDR	—	(T)	R	—	(T)	R	w S	(T)	R a	vw S ?	(T)	R a
	XD	—			—		w R28	—			—		(w) R60
30 minutes 625 C	EDT	—		R5 aaa	—		R15 aaa	—		R35 aaa	vw LPS ?		R60 aaa
	XD	—		R7	—		R10	—		R25	—	m R90	m R20
16 hours 625 C	EDT										LPS?		R70 aaa
	XD										—	w R85	R10

EDT — Electron diffraction in transmission (thin film)
EDR — Electron diffraction reflection (surface of film)
XD — X-ray diffraction (total film)
Rn — Rhombohedral phase: n ±10-15 mole percent Cr_2O_3 in αFe_2O_3-Cr_2O_3 solid solution.
RO — αFe_2O_3
LPS — Spinel, contains Fe and Cr or Ni, or both. Ni not to exceed 0.45 mole percent $NiFe_2O_4$.

m,w,vw — Medium, weak, very weak, respectively.
a — Oriented with (111) plane parallel to surface
b — Oriented with (110) plane parallel to surface
? — Orientation uncertain
(F) — EDR represents surface of base film
(N) — EDR represents nodules
(T) — EDR represents tops of nodules only

the metal against vacuum. The difference between the work functions of metal and oxide may also be of importance in determining 2. Since most of these factors can be affected by changes in the metal as well as by changes in the oxide, some of the micrographic experiments were made in a search for correlations between metal properties and the rate of nodule formation.

It was observed that when a film was stripped from the metal with Scotch tape and a second oxidation carried out, the positions of the new nodules bore no relation to those of the original ones. Thus if it is some local property of the metal which determines nodular formation, it must be a transient condition and not a permanent one such as its subgrain structure, position of slip bands, etc.

No significant effects of annealing and other heat treatments of the metal were observed other than those which could be ascribed to changes in distribution of chromium in the neighborhood of carbides.

Effects of Surface Preparation

The effects of surface preparation were noticeable in that a mechanical polish with 3/0 abrasive paper caused nodules to form preferentially along the scratches but the overall effect was much the same otherwise.

A metallographic polish, on the other hand, usually rendered the surface definitely more protective than did either the 3/0 or electrolytic polish. In other instances metallographically polished and 4/0 polished surfaces gave many nodules but they were much smaller than those occurring on an electrolytically polished surface. It is impossible to say whether these were real effects in the sense that the less perfectly crystallized, cold worked flow layer next to the oxide retarded breakdown of the film and growth of nodules, or whether the improvement was due to a change in the concentration of the alloying elements at the surface.

In order to check the possible effects of minor constituents of the alloys, specimens of the V series of nominal 26 percent Cr alloys shown in Table 1 were examined after 15, 45, 90 and 600 minutes of oxidation at 600 C. The alloys are arranged in the table in the order of increasing content of the minor constituents. There was a definite trend of variation of the attack with composition in the sense that those alloys low in the table gave fewer and generally smaller nodules. The fraction of film area covered by nodules was estimated to run from 10 percent at 15 minutes to 100 percent at 600 minutes for sample V-1, and the percentages decreased to 5 percent and 30 percent respectively for sample V-5. Thus, rather than being detrimental, as was anticipated, the minor constituents seem to reduce the rate of nodule formation.

A similar trend was noted in comparisons of AM-17 with the low carbon 19Cr-Fe sample K-19. It follows that the nodules do not seem to be caused by reduction of the film by carbon (or by silicon or manganese). Two specific correlations were noted. Samples V-2 and V-3 are nearly identical except V-3 has four times the manganese of V-2. Correlated with the smaller amount of manganese was the occurrence of much larger nodules, the number being roughly the same. To a lesser extent they were also larger on V-1 than on V-2. Thus the presence of manganese seems to reduce the rate of growth of the nodules after they have nucleated.

TABLE 4—Composition of Oxide Films on 26 Percent Cr-Steels With Variable Minor Constituents

Alloy	Method	OXIDATION TIME IN MINUTES AT 600 C			
		15	45	90	600
V-1 25.20Cr 0.016C 0.008Mn 0.14Si	EDT	w LPS R15	w LPS R40	— R50	w LPS (spots) R50
	XD	— RO	— m R25, RO	— w R25, RO	— w R75, RO
V-2 26.01Cr 0.022C 0.12Mn 0.16Si	EDT	— R15	m LPS R55	— R45	w LPS R30
	XD	m LPS RO	w LPS vw R50, RO	— vvw R75, RO	— vw R75, RO
V-3 26.20Cr 0.024C 0.55Mn 0.15Si	EDT	vvw LPS R25	— R50	— R50	— R40
	XD	vvw R100,RO	— w R50-RO	— m R75,RO	vw R75,RO
V-4 25.32Cr 0.027C 0.69Mn 0.50Si	EDT	vvw LPS R35	— R40	— R40	— R65
	XD	w R25,RO		— R50 (?)	— m m R75,R25
V-5 26.31Cr 0.14C 0.68Mn 0.74Si	EDT	vw LPS (br) R10	vw LPS (br) R40	— R50	vw LPS R60
	XD	— RO	vvw LPS m R50,RO		— m m R75,R25

EDT — Electron diffraction in transmission (thin film)
XD — X-ray diffraction (total film)
Rn — Rhombohedral phase: n ±10-15 mole precent Cr_2O_3 in αFe_2O_3-Cr_2O_3 solid solution.
RO — αFe_2O_3
LPS — Spinel, contains Fe and Cr or Ni, or both. Ni not to exceed 0.45 mole percent $NiFe_2O_4$.
m,w,vw — Medium, weak, very weak, respectively.
? — Orientation uncertain
(br) — Broad lines, small crystals or a range of composition

The films on both V-4 and V-5 were more protective than those on the other three alloys, which differ by having less silicon, in the following respects: For samples V-4 and V-5 there was less agglomeration of nodules after long oxidation times, less grain boundary trenching, less pitting of the base metal, and greater adherence of the base film to the metal, as shown in mechanical stripping experiments. Also the films were probably thinner. Thus the presence of silicon seems to affect the events which transpire in the metal as well as the adhesion between oxide and metal, and it may slow the growth process in the base film. Effects of this kind would be understandable if a silica layer were formed beneath the ordinary base film. All attempts to detect such a layer by diffraction have failed, but it may be present in the amorphous form.

Film Thickness

In connection with resistivity measurements of the oxide films formed on the AM series of chromium steels, Wiley[16] has determined their thickness versus time of oxidation at 600 C in air, using interferometric methods for the smooth films and weighing for the rough films. The curve for each alloy can be thought of as the sum of two curves. As the base film grows its thickness increases rapidly at first, then tends to flatten off, reaching a nearly constant value at 15 minutes of oxidation of approximately 400, 650, 700 and 900 Å on the AM-26, 17, 13 and 5 alloys, respectively. As the nodules start to form the average thickness begins to increase at an increasing rate. This latter behavior is to be expected from the general description given previously, since the number of nodules increases steadily and each continues to grow.

The thin film portions of these curves have the same general shape as the much more precisely determined ones for stainless steel measured by Gulbransen[17] using a continuous weighing microbalance. At his highest temperature of measurement (700 C) the stainless steel gave a rising "tail" similar to that for AM-13 or AM-17 at 600 C. This rise was attributed to "cracking"; the authors would attribute it to the formation of nodules, which of course may prove to be a consequence of cracking.

Oxide growth phenomena closely related to those discussed here have been observed on very pure iron under ordinary oxidation conditions at temperatures below 300 C[7,18] and at higher temperatures with a limited oxygen supply,[19,20,21] on nickel-rich Ni-Cr binary alloys,[22] on Ni[23] and on the (311) face of copper.[24] In all of these cases a thin oxide film and heavier local attack are observed.

The attack differs from that on the metals and under the conditions enumerated in Table 2 in two important respects. In the above cases the local growths are interpreted to be individual single crystals whereas under all of the conditions presently investigated the nodules are polycrystalline; large crystals are observed only after grain growth has progressed extensively. Secondly, with a limited oxygen supply the orientation of the crystallites, and/or the directions along which they nucleate, bear a pronounced relation to the orientation of the metal substrate. It is stated that this regularity disappears when the metal is not pretreated with a hydrogen anneal.[21] In none of the observations in Table 2, including those on hydrogen annealed specimens, have such directional effects been found.

It is evident from the variety of conditions under which it is observed that non-uniform oxidation is the rule rather than the exception. Not only is the phenomenon important in itself but discovery of any factor which has a specific effect on the nodular formation would give some much needed insight into the source of the base film's protectiveness. Furthermore, an understanding of the nodulated film growth may be of importance in interpreting the scaling process inasmuch as scaling does not start from the base metal, as is usually assumed, but is actually a continuation of the film growth which occurs during the initial heating period.

Composition of the Oxide Films

As has already been mentioned, the major constituent of the oxide films with which the authors are concerned is a rhombohedral phase. This phase may be accompanied by a minor spinel phase. Since the total range of lattice constants in the rhombohedral solid solutions of αFe_2O_3 and Cr_2O_3 is only about 1⅓ percent, and even smaller ranges occur in the spinels of interest, the uncertainty of approximately ½ percent which can be expected in routine transmission electron diffration measurements could not be tolerated.

The main sources of error in using the electron diffraction adaptor of the RCA EMU-2 electron microscope were found to be: (1) a drift of the accelerating voltage due to the temperature change of the voltage control resistor, (2) non-reproducibility of focusing controls, (3) non-uniformity of magnification over the field, and (4) non-reproducibility of photographic plate position.

The effects of these limitations were reduced by: (1) avoiding the first five minutes of operation, (2) use of a multiple specimen holder to decrease the time between photographs of sample and calibrating specimen, (3) calibration at several radii on the photographic plate and for each plate position. By taking these precautions, the uncertainty in composition of the rhombohedral phase was reduced to ± 10 to 15 mole percent Cr_2O_3, depending upon the quality of the photograph. The uncertainty of spinel lattice parameter was reduced to ± 0.008 to 0.01 Å for strong patterns.

The latter is not sufficient precision, unfortunately, to distinguish solutions of the Fe-Cr spinel system, but it is sufficient to differentiate three groups of Fe-Cr-Ni spinels when the pattern contains three or four lines not obscured by lines of the generally stronger rhombohedral pattern. Investigation of the lattice parameters of the spinels which may occur[1] shows that the three groups may be defined as follows: (1) spinel LPS (lattice parameter between 8.387 and 8.359 kx, contains Fe and Cr, or Ni not to exceed 0.45 mole fraction of $NiFe_2O_4$), (2) spinel LPS′ (parameter between 8.358 and 8.321

kx, contains Fe and Ni and may contain Cr), (3) spinel LPS″ (parameter between 8.320 and 8.290 kx, contains Ni and Cr and may contain Fe). Note: 1 kx unit = 1.00202A unit.

The oxidation in air of the AM series and other samples of chromium steels listed in Table 1 and Type 303 stainless steel were investigated by two or more of the diffraction methods over a range of temperatures and times. The results of these experiments are arranged in Tables 3-5.

Code Letters

The rhombohedral phases are indicated by R followed by a number indicating the mole percent of Cr_2O_3 in solution with Fe_2O_3; thus RO is Fe_2O_3 and R100 is Cr_2O_3. The spinels are indicated by the symbols just defined. For EDR data R and S are used for Rhombohedral and Spinel respectively, without attempting to specify the composition more closely. The relative amount of minor phases is indicated by m, w, vw over the type symbol to indicate medium, weak, very weak patterns, respectively. A letter is placed below the type symbol to indicate the type of fiber structure orientation, if any, which was observed; "a" and "b" refer to orientation with the (111) plane or the (110) plane, respectively, parallel to the metal surface. The number of such letters shows the degree of preferred orientation. The letter F, N, or T is placed in parenthesis at the bottom of the reflection data to indicate whether the primary contribution to the reflection diffraction pattern is made by the film (F), or the nodules (N), or by the tops of the nodules (T), as estimated from replica micrographs of the surfaces. This information is also useful in interpreting the X-ray diffraction data, for, with the usual sizes of nodules which are formed, the X-ray pattern of any films marked (N) or (T) will be determined very strongly by the material in the nodules. The base film contributes only a minor part.

Inspection of Table 3 of the AM series of chromium steels and Table 4 of 26-Cr steels with variable minor constituents, shows at once that the principal component of the base film (EDT) is a rhombohedral solid solution and that this may be accompanied by a relatively small amount of a spinel of the LPS type. In only one case is Cr_2O_3 observed, and the amount of it is very small. By a more careful examination of the EDT data it is evident that the Cr_2O_3 content of the principal component increases regularly with increase of either the time or the temperature of oxidation as well as with the chromium content of the alloy. The data suggest that during the initial stages of oxidation the ratio of chromium to iron is less in the base film then it is in the alloy.(1) At the other extreme of conditions the relative chromium content of the base film is several times that of the alloy, except for AM-5. It is evident that chromium enters the base film preferentially as oxidation proceeds. The observed variation of the composition and the observed rate of growth of the base film[16] are consistent with a nearly exclusive uptake of chromium after about the first five minutes of oxidation.

The rhombohedral solid solution in the base film is strongly oriented with the (111) plane parallel to the surface but with no indication of preferred

TABLE 5—Composition of Oxide Films on 18-8 Stainless Steel Type 303[1]

Time of Oxidation, Minutes	Method	OXIDATION TEMPERATURE 550 C	650 C	700 C
5	EDT	w LPS (b:) R30 a	vw LPS R15 a	w LPS″ R10 a
	EDR	— (F) R aaa	— (N) R aa	— (N) R a
	XD	— RO	vw LPS RO	w LPS RO
10	EDT			— R35 aa
	EDR			S R ? (T)
	XD			w LPS RO
15	EDT	— R35 a	vw LPS′ R35 ? aa	w LPS′ R35 ? aaa
	EDR	— R (N) aa	— R (N)	w S R ? (T)
	XD	— RO	vw LPS RO	m LPS RO
45	EDT	— R35 aa	w LPS′ R35 ? aa	
	EDR	— R (N) a	vw S R ? (N) (br)	
	XD	— RO	vw LPS RO	
105	EDT	LPS′ (spots) R35 aa	w LPS′ R45 aaa	
	EDR	— R (N)	w S R ? (N) (br)	
	XD	— RO	vw LPS RO	
705	EDT	— R50 aaa	w LPS′ R45 aaa	
	EDR	— R (N) (br)	w S R ? (T) (br)	
	XD	— RO	m LPS RO	
2145	EDT	— R50 aaa		
	EDR	— R (T) (br)		
	XD	— RO		

[1] 18.37Cr, 9.16Ni, 0.13C, 0.63Mn, 0.021P, 0.012S, 0.37Si.

EDT — Electron diffraction in transmission (thin film)
EDR — Electron diffraction reflection (surface of film)
XD — X-ray diffraction (total film)
Rn — Rhombohedral phase: n ± 10-15 mole percent Cr_2O_3 in αFe_2O_3-Cr_2O_3 solid solution
RO — Fe_2O_3
LPS — Spinel, contains Fe and Cr or Ni, or both. Ni not to exceed 0.45 mole percent $Ni Fe_2O_4$.
LPS′ — Spinel, contains Fe and Ni, may contain Cr.
LPS″ — Spinel, contains both Ni and Cr, may contain Fe.
m,w,vw — Medium, weak, very weak, respectively.
a — Oriented with (111) plane parallel to surface
b — Oriented with (110) plane parallel to surface
? — Orientation uncertain
(F) — EDR represents surface of base film
(N) — EDR represents nodules
(T) — EDR represents tops of nodules only
(br) — Broad lines, small crystals or a range of composition

(1) Since it is difficult to imagine that during the initial period of formation iron would be selectively oxidized in preference to chromium, the indicated trend for the relative chromium content of the film to be less initially than that of the alloy may mean that the metal surface is partially depleted of chromium in the electrolytic polishing process. Table 5 for 18-8 stainless steel would suggest that any such depletion is much less for this alloy than for the AM type alloys.

orientation in azimuth. In one run (30 minutes at 500 C) it was possible to re-mount the stripped films and obtain reflection diffraction patterns from their inside surfaces. These patterns showed that the inside of the film is not oriented, so that the orientation observed in the bulk of the base film must be determined by the direction of growth rather than by a direct influence of the metal.

Spinel Phase

In only a few cases was spinel observed in the base film. These occurred, with one exception, at the higher temperatures and on the 26 percent Cr alloys. The amount of the spinel was too small to permit determination of its orientation, if any.

The reflection diffraction results (EDR) show that the amount of spinel, relative to the rhombohedral structure, is considerably larger in the nodules than in the base film. The relative amount of spinel decreases with increasing nodular growth (i.e., to the lower left in each sub-table of Table 3). The decrease might be ascribed to a decrease in the accessibility to the beam of the inner portions of the nodules where the spinel might be expected to be located.

The same trend is noted, however, in the X-ray data of the various 26 percent Cr steels at 600 C. The LPS structure is found only in the shorter oxidation times and since all parts of the oxide are equally accessible to X-rays, it is concluded that the observed decrease in nodular spinel is real. The preferred orientation of this spinel is observed to decrease in degree along with the decrease in the relative amount of the phase, and is of two types; there is some indication that as growth proceeds the orientation changes from "b," (110) plane parallel to the metal surface, to "a" with the (111) plane parallel. The decrease in degree of preferred orientation of the spinel is accompanied by a decrease in the (111) type of orientation of the rhombohedral phase in the nodules. Any orientation determined by the plane of the metal or original base film surface would be expected to decrease as the nodule grows irregularly outward in all directions from its original starting point.

Rhombohedral Phase in Nodules

The X-ray data (XD) of the 26 percent Cr alloys for all but the shortest time of oxidation at 600 C, reveal two rhombohedral structures. The principal one is αFe_2O_3, or a low chrome solution the chromium content of which increases with the time of oxidation. The minor one is a high chrome solution the composition of which agrees, within the experimental error, with that of the base film. There can be little question that the low chrome phase is present in the nodules. According to Table 4 this phase is αFe_2O_3 on the more nearly pure binary alloys, but the presence of Si in the alloy (samples V-4, 5) correlates with the appearance of some Cr_2O_3 in solution with Fe_2O_3 after long times of oxidation.

For all of the exposure conditions for which X-ray data were taken, the nodular growth is well developed so that by far the majority of the X-ray patterns must be associated with the structure of the nodules. It follows that where samples AM-26 (Table 3) and samples V-4, 5, and possibly V-3 (Table 4), are exposed under conditions more severe than 15 minutes at 600 C the relative amount of the high chrome rhombohedral solution is much too large to be contained entirely in the base film. It must be assumed therefore that the nodules contain some high chrome solution not too different from that in the base film, in addition to the low chrome solution (or Fe_2O_3), and the spinel. Similarly, two solutions probably occur in the nodules formed on AM-5, 13, 17, but they are detected as only one phase because of limited resolving power. According to Table 4 the formation of the high chrome solution in nodules is favored by the presence of silicon, and possibly manganese, in the alloy.

Little information was obtained concerning the spacial arrangement of the components in the nodules except that the spinel probably is not on the outermost surface since it was observed by EDR to disappear with increasing time of oxidation. It may be that the high and low chrome rhombohedral solutions are present in different types of nodules.

Films on Type 303 Alloy

The formation of oxide films on Type 303, 18Cr-8Ni alloys in air has been studied extensively using all three diffraction methods correlated with micrographs. The results of this work are summarized in Table 5.

It will be seen for this alloy also that both the base film and the local surface growths have a rhombohedral phase as the principal component. There is even less spinel present than was found on the AM chromium steels for the comparable 550 C temperature. At the two higher temperatures small amounts of spinel do appear. Again, no Cr_2O_3 phase is observed. With this alloy the Cr_2O_3 content of the sesquioxide solution in the base film does not vary as widely as it does with the chromium steels. Its initial value is consistent with the alloy composition and quickly reaches a nearly constant value of 30 to 50 mole percent. The degree of (111) preferred orientation of this phase increases steadily during oxidation. In contrast with its properties in the base film, the rhombohedral phase in the nodules is shown by the X-ray data to be αFe_2O_3 (within 10 mole percent Cr_2O_3) at all times and temperatures. Also, the degree of the (111) preferred orientation of this oxide decreases steadily during growth.

At the two higher temperatures where they occur regularly, it is evident that the spinels are of two distinct types. EDT data give LPS′ or LPS″ in the base film (i.e., spinels containing iron, nickel and probably chromium), but the XD data give LPS (Fe, Cr, possibly Ni) spinels in the nodules. The amount of the latter spinels perhaps increases with time of oxidation. The amount of spinel was too small to permit determination of the preferred orientation. From these results it is clear that the oxide on the nickel-chromium steel differs from that on the chromium steels in that the spinel in the base film contains some Ni and the nodules do not include a high chrome rhombohedral phase.

Little analytical work has been done regarding the oxidation of these alloys when prepared by other means than electrolytic polishing, for the reasons previously stated. The work which has been done with mechanically polished surfaces indicates that the same general type of films are formed, but their spinel content is considerably higher and free metal has been found in some of them.

Summary

The formation of oxide films on chromium and nickel-chromium steels in the 500 C to 700 C range of oxidation in air may be described as follows:

A thin uniform film composed of small crystals of a ferric-chromic oxide solid solution forms very quickly with a [111] fiber axis normal to the surface. It may overlie a very thin layer of an Fe-Cr or Fe-Cr-Ni spinel type oxide. As time goes on the film increases in thickness at a decreasing rate and its chromium content increases. The crystal size increases and the preferred orientation becomes more nearly complete. During this growth of the base film its protectiveness fails at essentially randomly distributed points, whereupon a rapid local growth occurs in the form of polycrystalline nodules of αFe_2O_3, or of a low chrome Fe_2O_3-Cr_2O_3 solid solution, overlying a small amount of Fe-Cr spinel. A high chrome rhombohedral solution may also occur in the nodules. This phase is favored by the presence of silicon, but it is suppressed by 9 percent nickel in the alloy. These components are oriented but to a decreasing degree as growth continues.

Eventually the whole surface becomes covered with nodules. The frequency with which local failure occurs and the rate of growth after breakdown are decreased by a decrease in temperature and by an increase in the chromium content of the alloy. Small amounts of silicon and manganese also tend to reduce the rate of attack.

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Any discussions of this article not published above will appear in the December, 1957 issue.

Investigation of Inorganic Inhibitors for Minimizing Galvanic Corrosion of Magnesium Coupled to Aluminum*

By SARA J. KETCHAM and WALTER BECK

Introduction

THE BUREAU of Aeronautics established a project to develop materials that would inhibit the galvanic corrosion of magnesium when coupled with aluminum alloys. Inhibitors that appeared promising were to be used in the formulation of protective paint coatings.

The initial screening of inhibitors was conducted by a private laboratory under a BUAER sponsored contract.[1] That laboratory's results indicated that three inorganic inhibitors might prove worthwhile: magnesium vanadate ($Mg(VO_3)_2$), barium potassium chromate ($BaK_2(CrO_4)_2$), and calcium sulfide (CaS). Further study of these inhibitors at the Aeronautical Materials Laboratory indicated that all three of these compounds, when tested singly, reduced the galvanic corrosion current between AZ31B magnesium alloy and 2024-T3 aluminum alloy, as well as weight loss on magnesium in a 0.1 N NaCl electrolyte. Combinations of two or three of the inhibitors were even more effective.

In order to determine the mechanism of the inhibitor action with a view to improving it, the corrosion behavior of the aluminum and magnesium alloys were studied in separate compartments. Inhibitors were studied by adding them first only to the solution surrounding the magnesium, then only to the one with the aluminum, and finally to both solutions at the same time.

Data collected included corrosion current, polarization and recovery potentials, pH, and weight losses of both anode and cathode. This information was then compared with the results obtained in experiments with the electrodes in the same beaker.

Experimental Procedure

Isolated Electrode Studies

Panels of AZ31B-H24 magnesium alloy and 2024-T3 aluminum alloy, 5 inches x ½ inch x .064 inch, were used for this study. The panels were polished to a 240 grit alumina finish, scrubbed with pumice, rinsed with tap and then distilled water, dried with acetone, and weighed. The magnesium and aluminum alloys were placed in separate beakers and electrical contact was established by bridging with glass tubing filled with a jelly of agar in 0.1 N NaCl. The exposed area of the electrodes was 2¾ square inches, or .18 square decimeters.

The electrolyte was 0.1 N NaCl and specific amounts of inhibitor were added to 550 mls of solution. The

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Abstract

A fundamental study has been conducted of some inhibitors found to be effective in reducing galvanic corrosion between magnesium alloy AZ31B and aluminum alloy 2024-T3 in saline solutions. The object of the study was to determine the mechanism of protection afforded by the inhibitors, $Mg(VO_3)_2$, $BaK_2(CrO_4)_2$, and CaS, singly and in combination. The effect of these inhibitors on the polarization characteristics of the metals was determined with the electrodes isolated in separate compartments, bridging being accomplished with agar-NaCl bridges. In this group of tests, the polarization potentials, recovery potentials, and corrosion currents were measured, the latter by means of the zero resistance ammeter circuit. Additional tests were made with the electrodes in the same beaker, corrosion currents and corrosion rates being determined under these conditions.

Correlation of results from the potential, corrosion current, and corrosion rate data indicates that the $Mg(VO_3)_2$ and $BaK_2(CrO_4)_2$ polarize the aluminum strongly in the anodic direction, thereby reducing the galvanic current and the weight loss of magnesium.

Weight loss on aluminum was related to the increase in alkalinity occurring in experiments made in the same beaker.

Corrosion current measurements may serve as a useful tool for the determination of effectiveness of inhibitors, but the method should not be used indiscriminately.

Practical conclusions should only be drawn from the results obtained in experiments with both metals exposed in the same compartment. **5.8.3**

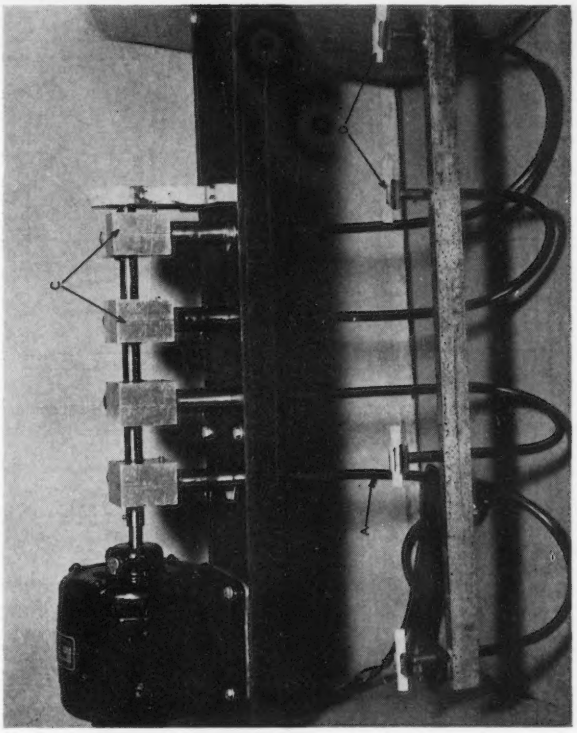

Figure 1—Stirring apparatus used in tests. Code to letters is as follows: A—flexible shafting; B—depth of immersion; C—gear boxes; D—magnetic stirring bars.

amounts used were based on the recommendation resulting from the initial screening and were as follows:

$Mg(VO_3)_2$	—1.3 gms
$BaK_2(CrO_4)_2$	—1.0 gms
CaS	—1.0 gms

The Mg $(VO_3)_2$ was completely soluble (concentration was 1.06×10^{-2} moles/1000 cc); the small quantities of $BaK_2(CrO_4)_2$ and CaS which go into solution are decomposed by hydrolysis. The $Mg(VO_3)_2$ solution was water clear. The solutions with addition of the other compounds were very turbid and contained small particles in suspension.

The exposure period was 96 hours, and the temperature 31.5 C ± 0.2. The solutions were constantly agitated. The stirring apparatus is illustrated in Figure 1. Standard magnetic stirring bars were placed in the beakers and driven by similar magnetic bars located beneath the beakers, rotated by flexible shafts encased in a tubular housing. The shafts are in turn operated by gears connected to a worm driven shaft, powered by an electric motor. The rate of stirring was 150 rpm.

After the runs were completed, the corrosion products were removed as follows:

Magnesium: 15 minutes immersion in a 20 percent CrO_3 solution containing 1 percent Ag_2CrO_4, at 212 F.

Aluminum: 10 minutes immersion at 180 F, in a solution containing 2 percent CrO_3 and 5 percent H_3PO_4.

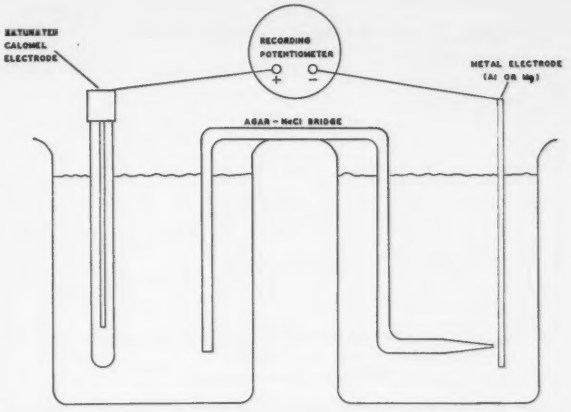

Figure 2—Method used to measure polarization potentials in isolated electrode experiments.

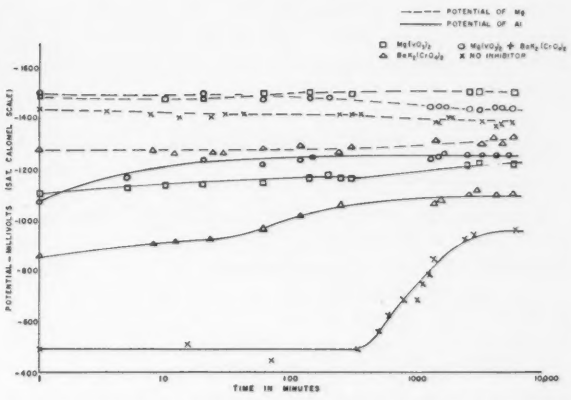

Figure 3—Effect of inhibitors on polarization potentials of aluminum and magnesium (isolated electrode experiments).

The potentials on both the aluminum and magnesium alloys were recorded, while current was flowing in the couple, utilizing the setup illustrated in Figure 2. In addition, the potential of each electrode, before coupling, was recorded until a steady state value was reached. The electrodes were then coupled for 96 hours. Following this, the electrodes were uncoupled and the degree of recovery to the original steady state potential value was recorded.[2]

Currents flowing between the magnesium and aluminum alloys were measured at various intervals by means of a zero resistance ammeter circuit.[3] In the latter stages of the investigation, the currents were recorded by means of two modified General Electric high speed recorders (Model CE) which gave exactly the same values as were obtained with the manually operated set-up.

The pH of all solutions was measured with a glass electrode at the beginning and end of each experiment.

Single Beaker Experiments

Experimental conditions were the same as those applied to the isolated electrode studies except the experiments were conducted at room temperature which was approximately 25 C. The distance be-

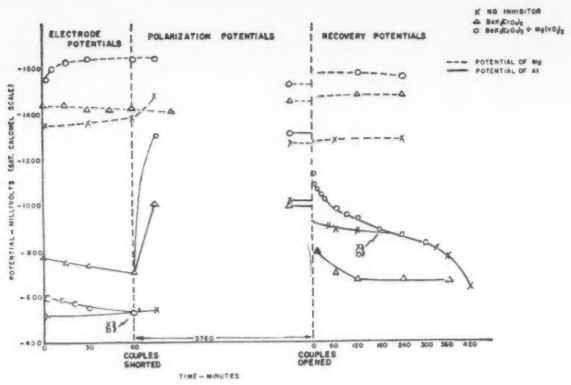

Figure 4—Determination of recovery potentials (isolated electrode experiments).

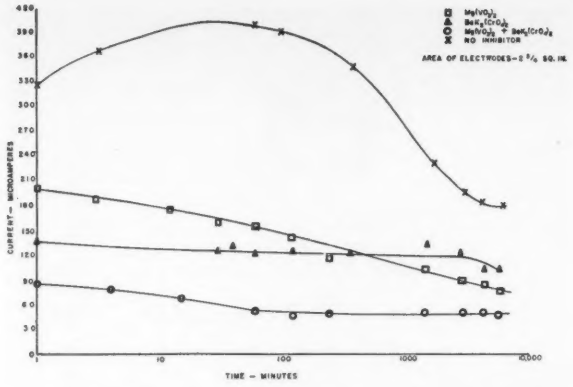

Figure 5—Corrosion currents—inhibitor in contact with aluminum (isolated electrode experiments).

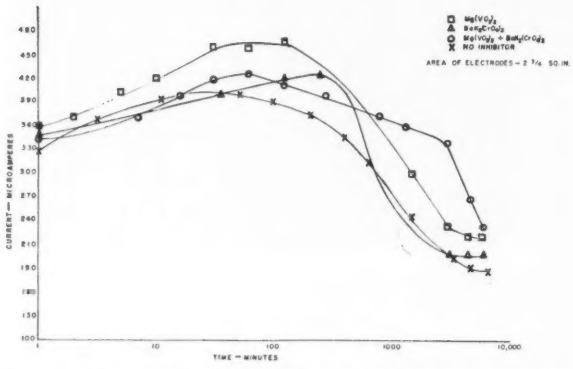

Figure 6—Corrosion currents—inhibitor in contact with magnesium (isolated electrode experiments).

tween the electrodes was 2⅛ inches. The instruments described above were also utilized for recording potentials and current in the single beaker equipment.

Experimental Results

Isolated Electrode Studies

Polarization potentials are graphically represented in Figure 3. Addition of the inhibitors shifts the potential of aluminum instantaneously and strongly in the anodic direction. $BaK_2(CrO_4)_2$ appeared also to have some effect on the potential of magnesium. To verify this, recovery potentials were measured. As shown in Figure 4, the differences between the potentials measured on magnesium prior to coupling and after uncoupling, are so slight they cannot be considered indicative of recovery. Therefore, any effect $BaK_2(CrO_4)_2$ might have on the potential of magnesium is considered to be negligible.

Potential measurements with CaS are omitted because reproducible results were not obtained. It appears that the CaS has some effect on the magnesium as well as the aluminum, but due to the instability of CaS, the exact nature of its effect cannot be determined.

Figure 5 presents the corrosion currents obtained when the inhibitors are added to the solution surrounding the aluminum electrode. These values are considerably less than the currents obtained in the uninhibited solution.

Figure 6 presents the currents obtained when the inhibitors are added only to the solutions surrounding the magnesium electrode. These currents are almost the same as those measured in the uninhibited solution.

Figure 7 shows that the currents obtained when the inhibitors are added to both solutions are approximately the same as those resulting when the inhibitor is added to the solution surrounding the aluminum electrode.

Weight loss measurements made with the electrodes in separate beakers connected by a salt bridge are considered to be of negligible value since the reproducibility is very poor. The highest weight loss obtained on the magnesium was 70 milligrams and on the aluminum 30 milligrams, both determined in the uninhibited solution after 96 hours exposure.

pH values are listed in Table 1. There is little or no increase in pH in the solutions containing the aluminum electrode, but increases were noted in the solutions containing the magnesium electrode with one exception ($BaK_2(CrO_4)_2$).

Single Beaker Experiments

Potentials were measured in the single beaker experiment in the uninhibited solution and in the solution containing the binary inhibitor system ($Mg(VO_3)_2$ and $BaK_2(CrO_4)_2$). The values and

TABLE 1—pH Values in Single Beaker and in Isolated Electrode Experiments

Single Beaker Experiment	No Inhibitor		$BaK_2(CrO_4)_2$		$Mg(VO_3)_2$		CaS		$Mg(VO_3)_2$ and $BaK_2(Cr_4O)_2$	
Initial pH	7.7		8.8		7.5		10.7		7.7	
Final pH (after 96 hours)	11.4		10.9		10.0		10.9		9.7	
Isolated Electrode Experiments	**Mg**	**Al**	**Mg**	**Al**	**Mg**	**Al**	**Mg**	**Al**	**Mg**	**Al**
Initial pH	7.7	7.7	8.8	8.8	7.5	7.5	10.7	10.7	7.5	7.5
Final pH (after 96 hours)	9.4	7.8	8.8	8.8	9.1	7.7	11.2	11.2	9.2	7.9

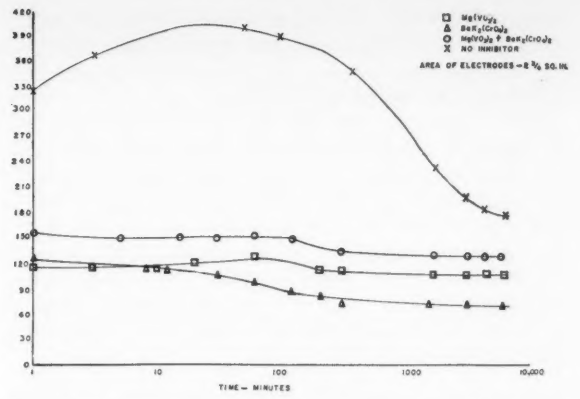

Figure 7—Corrosion currents—inhibitor in contact with both electrodes (isolated electrode experiments).

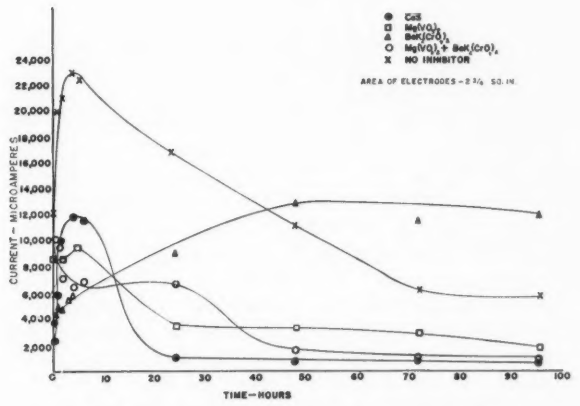

Figure 8—Corrosion currents—electrodes coupled in same beaker.

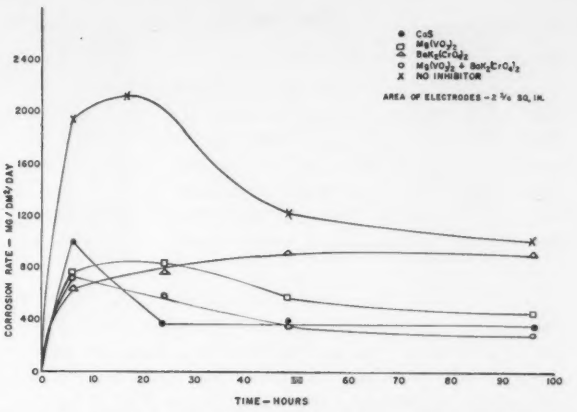

Figure 9—Corrosion rate of magnesium in single beaker experiments.

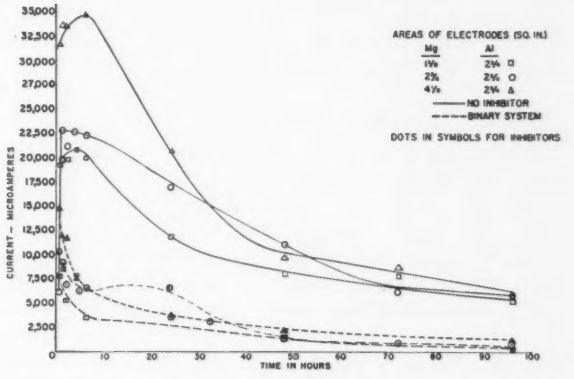

Figure 10—Effect of change of anode area on corrosion currents.

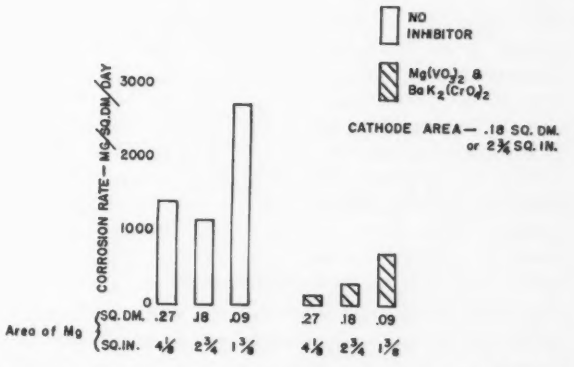

Figure 11—Effect of change of anode area on corrosion rate. Duration of test was 96 hours.

trends of the potentials were similar to those obtained in the isolated electrode studies.

Corrosion currents measured are shown in Figure 8. With the exception of the binary system, the currents increase during an initial period, but later decrease with time. The values given are the average of three runs and the maximum deviation after the steady state condition is reached does not exceed 15 percent.

As shown in Table 2, the weight losses (and pitting attack) on magnesium were decreased by the addition of inhibitors, the binary system being the most effective. Weight loss on the aluminum, however, was decreased only by $BaK_2(CrO_4)_2$ and combination of this compound with $Mg(VO_3)_2$. CaS and $Mg(VO_3)_2$ increased the weight loss on the aluminum. The weight losses listed are the average of at least three determinations and the reproducibility was within 8 percent. The uncoupled weight losses are small in all cases. The corrosion rates for magnesium depicted in Figure 9 decreased with time in all cases except that of $BaK_2(CrO_4)_2$. Finally, a steady state was approached in all cases.

The data compiled in Table 1 shows that the pH always increases with time, the largest increase being noted in the uninhibited solution and in the solution containing $Mg(VO_3)_2$.

The effectiveness of the inhibitors was further investigated by varying the anode area in relation to the cathode area. The latter was kept constant and the anode area was reduced by 50 percent and then increased by 50 percent. The current values obtained are shown in Figure 10 and the weight loss values in Figure 11.

Discussion

Mechanism of Action of Inhibitors

Magnesium exposed in a separate container without inhibitor addition, maintains the same potential throughout the entire exposure period while the trend on the corresponding potential time curve recorded on aluminum is strikingly similar to that of the corrosion current. These results clearly indicate that the galvanic corrosion of magnesium is under cathodic control.

Addition of the inhibitors to the solutions surrounding either the aluminum or magnesium or both, does not affect the potential of the magnesium nor does addition of the inhibitor to the solution with the magnesium electrode affect the corrosion current. However, the rapid and marked shift of the potential of aluminum in the anodic direction, occurring when chromate or vanadate or both are present in the solution surrounding the aluminum, proves that it is cathodic polarization which causes the marked depression of the corrosion current.

Corrosion Current and Corrosion Rate

A discussion of the extent to which corrosion current can be correlated with the corrosion rate of magnesium, will be based on the results obtained with magnesium coupled to aluminum in the same container. The similarity of the trends of the rate and current-time curves recorded in uninhibited solutions, or in solutions with CaS, $Mg(VO_3)_2$, or the chromate and vanadate combination added is striking. A marked depression of the current coincides with a strong retardation of the corrosion rate. At first glance there appears to be a discrepancy between current and rate in the $BaK_2(CrO_4)_2$ solution. The rate curve is below, and the current curve is above, that resulting in the uninhibited solution, after a period of 40 hours was exceeded. The difference in magnitude between the corrosion currents during the first 40 hours is great but within the last 56 hours it is comparatively small. Therefore, the corrosion rate on magnesium in the uninhibited solution is, as it should be, much greater within the first 40 hours than it is during the last 56 hours in the inhibited solution. The corrosion current in the $BaK_2(CrO_4)_2$ solution never becomes great enough to cause the rate to exceed that in the uninhibited solution.

In acordance with the findings of other authors, there was no agreement between the experimentally determined weight loss of magnesium and that calculated from Faraday's law,[4] the latter being lower.

Effect of Combination of Inhibitors

The effect of combining the $Mg(VO_3)_2$ and $BaK_2(CrO_4)_2$ is to improve the performance of both. Neither the current nor the weight loss values for the magnesium are an average of the values obtained when the inhibitors are used singly, but are lower than in either case. The reason for this improvement is not clear. Some complex chemical reaction may take place which results in a coating that polarizes the local cathodes on the aluminum more effectively than do the single inhibitors.

Area Experiments

Figures 10 and 11 show that the effectiveness of the inhibitor system is not influenced by change of area of the magnesium, and therefore, that the corrosion is inhibitor controlled.

Behavior of Aluminum in NaCl Solutions Without and With Addition of $Mg(VO_3)_2$

Aluminum suffered considerable weight loss, believed to be due to the increased alkalinity of the solutions as a result of the formation of $Mg(OH)_2$. The pH in the uninhibited solution was 11.4 after 96 hours, while that of the solution with $Mg(VO_3)_2$ present was 10. According to Groot and Peekema[5] these pH values fall within the range of so-called "caustic corrosion" of aluminum.

Even though the solutions were agitated, it is considered possible that the rate of agitation was not sufficient to remove all of the sodium hydroxide formed at the cathode. Therefore, the pH at that region might be even higher than in the bulk of the solution, and is definitely greater in the case of experiments in one compartment than in two compartments due to the higher current density on the cathode.

Evidently, in the experiments made in one compartment, two processes affect the aluminum:

1. A polarization resulting finally in an inhibition of the corrosion of magnesium.
2. An attack caused by alkalinization.

In the experiments in two compartments, only the first process occurs. The pH was approximately 8 in the solutions containing the aluminum electrode because there was no formation of $Mg(OH)_2$ and, therefore, no measurable weight loss on this metal.

TABLE 2—Weight Loss Determinations in Single Beaker Experiments[1]

Time (Hours)	Condition	No. Inhibitor Wgt. Loss (mgms)		$BaK_2(CrO_4)_2$ Wgt. Loss (mgms)		$Mg(VO_3)_2$, Wgt. Loss (mgms)		$BaK_2(CrO_4)_2$ + $Mg(VO_3)_2$, Wgt. Loss (mgms)		CaS, Wgt. Loss (mgms)	
		Mg	Al	Mg	Al	Mg	Al	Mg	Al	Mg	Al
6	Coupled	88.0	18.6	29.3	8.8	35.0	18.0	34.2	20.6	45.0	84.5
16½	Coupled	257.0	50.2	....	....	....	....	....	....	....	....
24	Coupled	....	....	138.0	36.6	150.0	94.0	106.0	81.9	67.2	112.5
48	Coupled	439.0	98.3	330.1	95.5	206.0	181.0	135.0	107.0	145.3	163.8
96	Coupled	732.0	198.8	662.1	145.0	331.8	243.7	213.0	185.0	261.3	323.9
24	Uncoupled	9.8	16.3	2.9	0.1	27.9	0.5	23.0	0.8	....	....
48	Uncoupled	12.5	19.0	3.3	0.8	37.7	0.8	24.5	0.4	....	....
72	Uncoupled	20.0	26.0	3.1	0.0	38.0	0.5	30.0	0.3	....	....
96	Uncoupled	25.8	30.0	6.8	0.0	36.0	0.5	32.0	0.4	11.2	221.3

Note: Exposed area was 2¾ square inches.
[1] Average of at least three runs.

These findings show clearly that for establishing the mechanism which controls the inhibitor action, experiments in separate compartments are significant, or even indispensable. The fact that the aluminum is corroded when the electrodes are in the same container is of great practical importance; therefore, practical conclusions should only be based on the results obtained in one compartment.

Behavior of Aluminum in NaCl Solution in Presence of $BaK_2(CrO_4)_2$

Although the solution containing $BaK_2(CrO_4)_2$ had a final pH of 10.9, the weight loss on the aluminum was the lowest obtained. This result is in accordance with that of McKee and Brown,[6] who found that chromates effectively inhibited the corrosion of commercially pure aluminum (1100) in alkaline solutions. The mechanism differs in the two cases, however. In the case of McKee and Brown, the protection is due to the comparatively large quantity of soluble chromate (500×10^{-3} moles/1000 cc) which causes anodic polarization. The amount of soluble chromate ($CrO_4^=$) formed and maintained in the experiments with $BaK_2(CrO_4)_2$ containing solutions is only 4×10^{-3} moles/1000 cc. An extremely adherent yellow coating forms on the surface of the aluminum, probably by adsorption from the suspension. The appearance of this coating is shown in Figure 12. By X-ray diffraction, chromate compounds were identified in this coating.

According to Kastens and Prigotsky,[7] when $BaK_2(CrO_4)_3$, being a true double salt, is incorporated into paint pigments, it hydrolyzes slowly when the paint film is exposed to moisture, and releases K_2CrO_4, leaving $BaCrO_4$ behind as a residual coating with substantial protective properties. In this case, therefore, it is believed that the protective action of the $BaK_2(CrO_4)_2$ is due to the residual insoluble chromate coating which forms on the aluminum surface and protects the metal by acting as a diffusion barrier. This coating does not form on aluminum when the electrodes are in separate compartments, illustrating again that a complete appraisal can not be made on the basis of the isolated electrode studies alone.

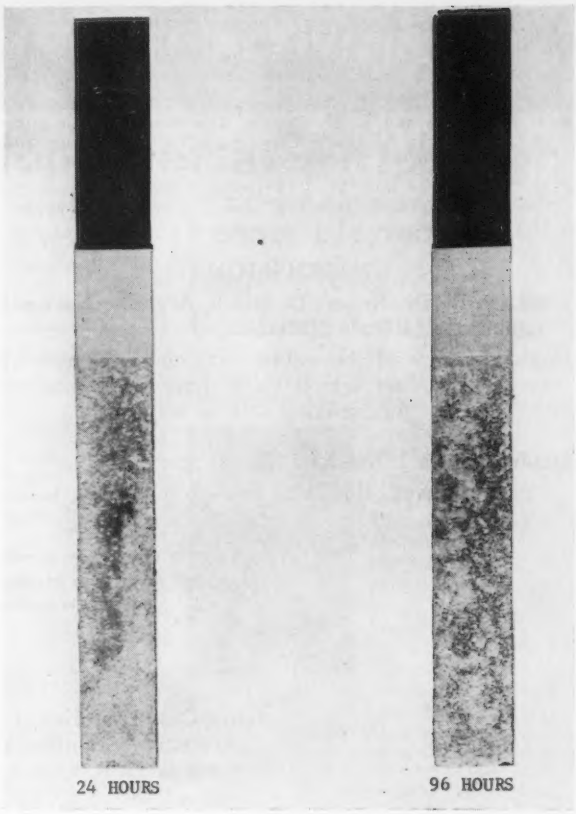

Figure 12—Appearance of coating formed on aluminum in solution containing $BaK_2(CrO_4)_2$.

Conclusions

The corrosion of magnesium when coupled with aluminum is under cathodic control. The effect of the inhibitors used in this study is to polarize the aluminum instantaneously and markedly in the anodic direction, thereby retarding the corrosion rate.

Measurements of the corrosion current correlate with the corrosion rate.

Isolated electrode studies are helpful in determining mechanism, but practical conclusions should be based only on experiments conducted in a single container.

A combination of $Mg(VO_3)_2$ and $BaK_2(CrO_4)_2$ are effective in retarding the corrosion of magnesium coupled to aluminum in aqueous salt solutions. Their efficacy in, and compatibility with, other media such as paints is now under investigation and will be reported later. Due to its instability and offensive odor, CaS is considered unsatisfactory for use as an inhibitor.

NOTE: The opinions or assertions expressed in this article represent only the personal views of the writers, and are not to be construed as official or reflecting the views of the Department of the Navy or the Naval Service at large.

Acknowledgment

The authors are much indebted to F. S. Somers and J. H. James of the Bureau of Aeronautics, F. S. Williams, Superintendent, Metallurgical Division, Aeronautical Materials Laboratory, and H. A. Robinson, Assistant Head of the Metallurgical Laboratory, Dow Chemical Company, for the great interest taken in this study.

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DISCUSSION

Question by Dr. Robert D. Misch, Argonne National Laboratory, Lemont, Illinois:

Could the high pH be caused by a charged colloidal corrosion product which could have an excess of Al^{+++} or Mg^{++} while leaving OH^- in solution?

Reply by Sara J. Ketcham and Walter Beck:

It is believed that the corrosion products are largely insoluble rather than in colloidal distribution. This does not exclude, however, the possibility that a surplus of OH^- may exist in the solution, and be partly responsible for the high alkalinity. The Metallurgical Laboratory of the Dow Chemical Company* has, by X-ray diffraction of the insoluble corrosion products formed in an aluminum-magnesium cell, identified the compound magnesium oxychloride ($5\,Mg(OH)_2 \cdot MgCl_2 \cdot 7H_2O$). The assumption has been made that in addition to the formation of $Mg(OH)_2$ the following reaction proceeds: $6Mg^{++} + 10\,OH^- + 2Cl^- + 7H_2O = 5Mg(OH)_2 \cdot MgCl_2 \cdot 7H_2O$.

This reaction requires the removal of ten hydroxyl ions instead of twelve for every six magnesium ions. The two additional OH^- ions which remain in solution can account for the increase of pH without requiring a colloidal distribution of the corrosion products.

* Private communication.

Any discussions of this article not published above will appear in the December, 1957 issue.

ERRATA

Some Case Histories of Stress Corrosion Cracking of Austenitic Stainless Steels Associated with Chlorides by H. R. Copson and C. F. Cheng. Corrosion, Vol. 13, No. 6, 397t-404t (1957) June.

On page 398t the caption for illustration identified as Figure 2 should read as follows:

Figure 7—Cross section through stainless clad steel of Case 7 showing cracks stopping at the steel. Nital etch, 25X.

On page 399t the captions for illustrations identified as Figures 3, 4, 5 and 6 should read respectively:

Figure 8—Section through expansion joint of Case 10 showing cracking at inside surface. Electrolytic oxalic acid etch, 250X.

Figure 9—One of the cracked disc specimens of Case 11.

Figure 4—Part of screw conveyor of Case 13 showing cracks. ½X.

Figure 10—Part of flash shield of Case 14 showing cracks. 1X.

On page 400t the captions for illustrations identified as Figures 7 and 8 should read respectively:

Figure 2—Cross section of flash shield of Case 14 showing cracks. Electrolytic oxalic acid etch, 100X.

Figure 5—One of the cracked disc specimens of Case 15. 1X.

On page 402t the captions for illustrations identified as Figures 9 and 10 should read respectively:

Figure 6—Cracking in bottom of kettle of Case 17. 1X.

Figure 3—Cross section of stainless steel of Case 21 showing cracking. Etched with glyceregia, 100X.

Record Committee Sessions Set at Pittsburgh

Northeast Region Schedules Eleven Meetings Nov. 12

A record number of committee meetngs will be held at the Northeast Region Meeting scheduled November 12-14 at the Penn-Sheraton Hotel in Pittsburgh. Eleven will be held Tuesday, November 12, a day set aside exclusively for technical committee meetings. Committees that will meet and subjects to be discussed are as follows:

Unit Committee T-2E Internal Corrosion of Product Pipe Lines and Tanks will meet at 9:30 am. T-2E will discuss reports on the product pipe line in-line corrosion coupon tests from participating compaines.

Unit Committee T-2K Prefabricated Plastic Film for Pipe Line Coating will meet at 9:30 am. A suggested minimum requirements specification for prefabricated plastic film will be presented and discussed at the meeting. It is expected progress reports also will be heard from task groups on research and development and history and results.

Unit Committee T-3C Annual Losses Due to Corrosion will meet at 2 pm. T-3C expects to hear a report on a questionnaire covering use of coating materials, paint mastics and metals or non-metals corrosion preventive materials in corrosion environments.

Unit Committee T-3H Tanker Corrosion will meet at 2 pm. T-3H will hold an open forum type discussion on methods for mitigating tanker corrosion.

Unit Committee T-4B Corrosion of Cable Sheaths will meet at 9:30 am. Progress by task groups on cathodic protection, tests and surveys, protection of pipe type cables, non-metallic sheaths and coatings, lead and other metallic sheaths and stray current electrolysis will be discussed.

Task Group T-5A-5 Nitric Acid Corrosion will meet at 9:30 am. T-5A-5 expects to discuss results of a survey on corrosion by nitric acid.

Task Group T-5A-6 on HF Corrosion will meet at 2 pm to discuss HF corrosion and hear a report from a work group.

T-6D Industrial Maintenance Painting will meet at 2 pm to hear reports from task groups on economics of maintenance, standardization of scope of painting specifications and paint programs.

Unit Committee T-6F Protective Interior Linings, Applications and Methods will meet at 9:30 am to hear progress reports from task groups on curing, surface preparation, inspection, safety and application.

Unit Committee T-5D Plastic Materials of Construction will meet at 9:30 am to discuss a report on the use of plastic materials of construction in the chemical process industries. Also to be discussed is the plastics numbering system being devised by Task Group T-5D-7 on Engineering Design.

Unit Committee T-7A Northeast Region Corrosion Coordinating Committee will meet at 2 pm. T-7A will discuss problems of corrosion coordinating committees and electrolysis committees with emphasis on establishing liaison among coordinating committees.

TECHNICAL COMMITTEE MEETINGS
NOVEMBER 12, 1957

NORTHEAST REGION MEETING
Nov. 12-14, 1957
Penn-Sheraton Hotel, Pittsburgh

Morning

- **T-2E** Internal Corrosion of Products Pipe Lines and Tanks
- **T-2K** Prefabricated Plastic Film for Pipeline Coatings
- **T-4B** Corrosion of Cable Sheaths
- **T-5A-5** Corrosion by Nitric Acid
- **T-5D** Plastic Materials of Construction
- **T-6F** Protective Interior Linings Application and Methods

Afternoon

- **T-3C** Cost of Corrosion
- **T-3H** Tanker Corrosion
- **T-5A-6** HF Corrosion
- **T-6D** Industrial Maintenance Painting
- **T-7A** Northeast Region Corrosion Coordinating Committee.

Biological Deterioration Group Elects Richards

A. P. Richards of the Wm. F. Clapp Laboratories at Duxbury, Mass. has been appointed vice-chairman of NACE Group Committee T-9 on Marine Biological Deterioration. The appointment was made by E. C. Greco, United Gas Corporation, Shreveport, La. chairman of the Technical Practices Committee shortly after the T-9 organization meeting held at the Seahorse Institute at Wrightsville Beach, North Carolina.

At Wrightsville Beach three unit committees were proposed by the group. One of these is to be known as biological, a second preservatives and their appraisal, and a third practical applications.

Dr. Charles Lane of the University of Miami was appointed tentative chairman of the group to be known as the Biological Committee.

Dr. Tom Sweeny of the U. S. Naval Research Laboratory was appointed tentative chairman of the proposed unit committee on preservatives and their appraisal.

A tentative chairman for the third Unit Committee will be appointed later.

Fenner Elected to Head Protective Coatings Committee

Otto H. Fenner, Monsanto Chemical Co., St. Louis, Mo. is the newly elected vice-chairman of Group Committee T-6 on Protective Coatings. Results of the July election were announced by the chairman of the committee, L. S. Van Delinder, Union Carbide Chemicals Company, Division of Union Carbide Corp., South Charleston, W. Va.

Fenner

Mr. Fenner, a member of NACE since 1947, has been active in association work. He was general conference chairman of the 13th Annual NACE Conference and 1957 Exhibition. He is co-author of a paper, "The Engineering Properties of Rigid Polyvinylchloride" presented at the 1957 Conference and is co-author of an article, "Design and Engineering of Plastic Materials of Construction."

In addition to being vice-chairman of T-6, Mr. Fenner is chairman of Task Group T-5D-7 on Engineering Design (Plastic Materials of Construction); and is a member of the following committees and task groups: T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion; T-6A-17 on Polyurethanes (Organic Coatings and Linings for Resistance to Chemical Corrosion); and T-6D on Industrial Maintenance Painting.

Coordinating Group's Activity Is Growing

Group Committee T-7 on Corrosion Coordinating Committees continues to grow under the chairmanship of C. A. Erickson, Jr., Peoples Natural Gas Company, Pittsburgh. The committee is sponsoring and will present a symposium on Underground Corrosion Coordination at the annual conference to be held in San Francisco next March. The Western Pennsylvania Corrosion Committee under T-7A Northeast Region Corrosion Coordinating Committee will present a symposium on Underground Corrosion Coordinating at the Northeast Region meeting, Pittsburgh, on November 12-14.

Since the committee's organization at New York in March 1956, five unit committees corresponding with the regional divisions of the Association have been organized. Officers of the Unit Committees are as follows:

Unit Committee T-7A Northeast

(Continued on Page 84)

Technical Program and Committee Meetings, Oklahoma City, Oct. 1-4

MON.	P.M.	**T-1B** Condensate Well Corrosion					
TUESDAY	A.M.	**T-3D-1** Electrical Holiday Inspection of Coatings	**T-2J** Wrappers for Underground Pipe Line Coating	**T-2D** Standardization of Procedures for Measuring Pipe Coating Leakage Cond.	**T-8** Corrosion of Petroleum Refinery Equipment	**T-6A** Organic Coating & Linings for Resistance to Chemical Corrosion	**T-1** Corrosion In Oil & Gas Well Equipment
	P.M.		**T-2K** Prefabricated Plastic Film for Pipe Line Coating	**T-6B** Protective Coatings for Atmospheric Corrosion	**T-5C-1** Corrosion by Cooling Waters	**T-2C** Minimum Current Requirements	**T-1** 1:30 to 3:30 3:30 **T-1D** Sour Oil Well Corrosion
WEDNESDAY	A.M.	**T-5E** Stress Corrosion Cracking of Austenitic Stainless Steel	**T-1F** Metallurgy **T-1F-1** Sour Stress Crack **T-1F-2** Sweet Stress Crack	**T-2F** Internal Corrosion of Crude Oil Pipe Lines & Tanks	**T-6E** Protective Coatings in Petroleum Production	**T-1K** Inhibitors for Oil & Gas Wells	**T-3G** Cathodic Protection
	P.M.		**T-2B** Anodes for Impressed Current	**T-1H** Oil String Casing Corrosion	**T-7D** South Central Region Corrosion Coordinating Committee	**T-6D** Industrial Maintenance Painting	**T-8** Corrosion of Petroleum Refinery Equipment
THURSDAY	A.M.				**Marine Corrosion Symposium**	**Process Industry Symposium**	**Corrosion in the Gas Distribution Industry Symposium**
	P.M.			**T-1M** Corrosion of Oil & Gas Well Prod. Equipment in Offshore Installations		**Process Industry Symposium**	**Cathodic Protection Symposium**
FRI	A.M.					**Proctective Coatings Symposium**	**Oil & Gas Production Symposium**

Coordinating Group's—

(Continued From Page 83)

Region—C. A. Erickson, Jr., Peoples Natural Gas Company, Pittsburgh, Pa., chairman; L. Andrew Kellogg, Niagara Mohawk Power Company, Syracuse, New York, vice-chairman.

T-7B North Central Corrosion Coordinating Committee—Paul C. Hoy, Dayton Power & Light Company, Dayton, Ohio, chairman; J. O. Mandley, Consolidated Gas Company, Detroit, vice-chairman.

Unit Committee T-7C Southeast Region Corrosion Coordinating Committee—Ernest W. Seay, Jr., Chesapeake & Potomac Telephone Company of Virginia, Norfolk, chairman; R. W. Hamel, Commonwealth Natural Gas Corporation, Richmond, vice-chairman.

Unit Committee T-7D South Central Region Corrosion Coordinating Committee—O. W. Wade, Transcontinental Gas Pipe Line Corp., Houston, chairman; C. L. Woody, United Gas Corporation, Houston, vice-chairman.

Unit Committee T-7E Western Region Corrosion Coordinating Committee—Irwin C. Dietze, Department of Water & Power, Los Angeles, chairman; J. S. Dorsey, Southern California Gas Company, Los Angeles; vice-chairman.

Unit Committee T-7F Canadian Region Corrosion Coordinating Committee—C. L. Roach, Bell Telephone Company of Canada, Montreal, Quebec, chairman; R. E. Kuster, Union Gas Company of Canada, Ltd., Chatham, Ontario, vice-chairman.

C. L. Mercer, Southwestern Bell Telephone Company, Houston, is vice chairman of Group Committee T-7.

Since meeting held in St. Louis, eight coordinating committees have affiliated with the T-7 organization. Six of these are under T-7A Northeast Region Corrosion Coordinating Committee. They are:

1. Charleston Coordinating Committee on Corrosion.
2. Greater Boston Electrolysis Committee.
3. Western Pennsylvania Corrosion Committee
4. Pittsburgh Corrosion Committee
5. New Jersey Committee on Corrosion
6. Greater New York Committee on Corrosion

Affiliated under Unit Committee T-7B on North Central Region Corrosion Coordinating Committee is the Chicago Section Committee on Underground Corrosion.

Houston Corrosion Coordinating Committee has affiliated with Unit Committee T-7B South Central Region Corrosion Coordinating Committee.

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Anode size: 4" x 80" "National" graphite (plain)
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Wide Variety of Topics to Be Discussed at Oklahoma City Technical Committee Sessions

Subjects to be discussed at the 28 technical committee meetings to be held during the South Central Region meeting in Oklahoma City are: **T-1 Corrosion of Oil and Gas Well Equipment,** Tuesday, October 1 from 9 am to 3:30 pm. Progress reports from T-1 unit committees will be discussed during the morning session of the T-1 meetings. Afternoon sessions will be devoted to new developments and preventive measures in oil and gas well equipment. Unit committee chairman are being urged by T-1 chairman, Jack L. Battle of Humble Oil & Refining Company, Houston, to prepare comprehensive progress reports of problems that have been considered since the meetings in St. Louis.

Unit Committee T-1B Condensate Well Corrosion, Monday, September 30 at 2 pm. T-1B recently has been primarily concerned with what is sometimes termed as "super" high pressure gas condensate wells. Specifically the committee has been considering wells with shut-in pressures that range from 7000 to 12,000 psi. A task group was appointed at the St. Louis meeting to consider the problem of high pressure well completion and corrosion mitigation procedures. The task group, under the chairmanship of W. F. Oxford, Jr., Sun Oil Company, Beaumont, Texas has held two meetings since the conference in St. Louis. A questionnaire has been distributed and results tabulated. It is expected that the task group will have considerable information to present to the Unit Committee at Oklahoma City.

Unit Committee T-1D Sweet Oil Well Corrosion, Tuesday, October 1, 3:30 pm. The T-1D task group on pump corrosion is expected to report on the possibility of preparing a chart showing interchangeable pump parts similar to the one used in oil well sucker rods. The group also has been attempting to learn whether producing companies have sufficient data to show what pump parts have a record of high percentage of failures and if possible, to determine whether the failures are due to corrosion or other causes.

Other subjects to be discussed at the T-1D meeting include field testing of inhibitors being injected into oil producing formations; plastic coating for tanks and lines and general methods of inhibiting oil well corrosion.

Unit Committee T-1F on Metallurgy, October 2, 9 am. The T-1F meeting will be followed immediately by a meeting of the T-1F-1 task group on Sulfide Stress Corrosion Cracking. The T-1F-1 meeting will be followed by a meeting of T-1F-2 on Sweet Crude and Sweet Condensate Corrosion Cracking.

Unit Committee T-1H on Oil String Casing Corrosion, Tuesday, October 2, 2 pm. The four T-1H task groups have been concentrating on summarizing casing corrosion control methods used throughout the country. The committee has developed information on various procedures used over the past few years and now is attempting to get actual data. Data to be discussed at the meeting include the number of companies actually in need of a corrosion control program, kind of protection being used, when the program was initiated, number of wells under protection in the area, criteria used to determine protective measurements, estimated effectiveness of control program to date and the estimated cost of protecting individual wells.

Unit Committee T-1K on Inhibitors for Oil and Gas Wells, Wednesday, October 2, 9 am. T-1K will hear reports from task groups on sour crude inhibitors, evaluation and sweet crude inhibitor evaluation. The T-1K proposed laboratory procedure for screening inhibitors for use in oil and gas wells will be discussed.

Unit Committee T-1M, Corrosion of Oil and Gas Well Equipment in Offshore Installations, Thursday, October 3, 2 pm. T-1M will present a symposium on marine corrosion on Thursday morning. It is expected the meeting will be devoted to papers presented at the symposium and other developments in the protection of oil and gas well equipment in offshore installations.

Unit Committee T-2B on Anodes for Impressed Current, Wednesday, October 2, 2 pm. Progress reports will be heard from task groups on sources of impressed current for anodes, ground bed design data, use of high silicon cast iron for anodes and use of platinum, palladium and other metals for anodes.

Unit Committee T-2C on Minimum Current Requirements, Thursday, Octo-

(Continued on Page 88)

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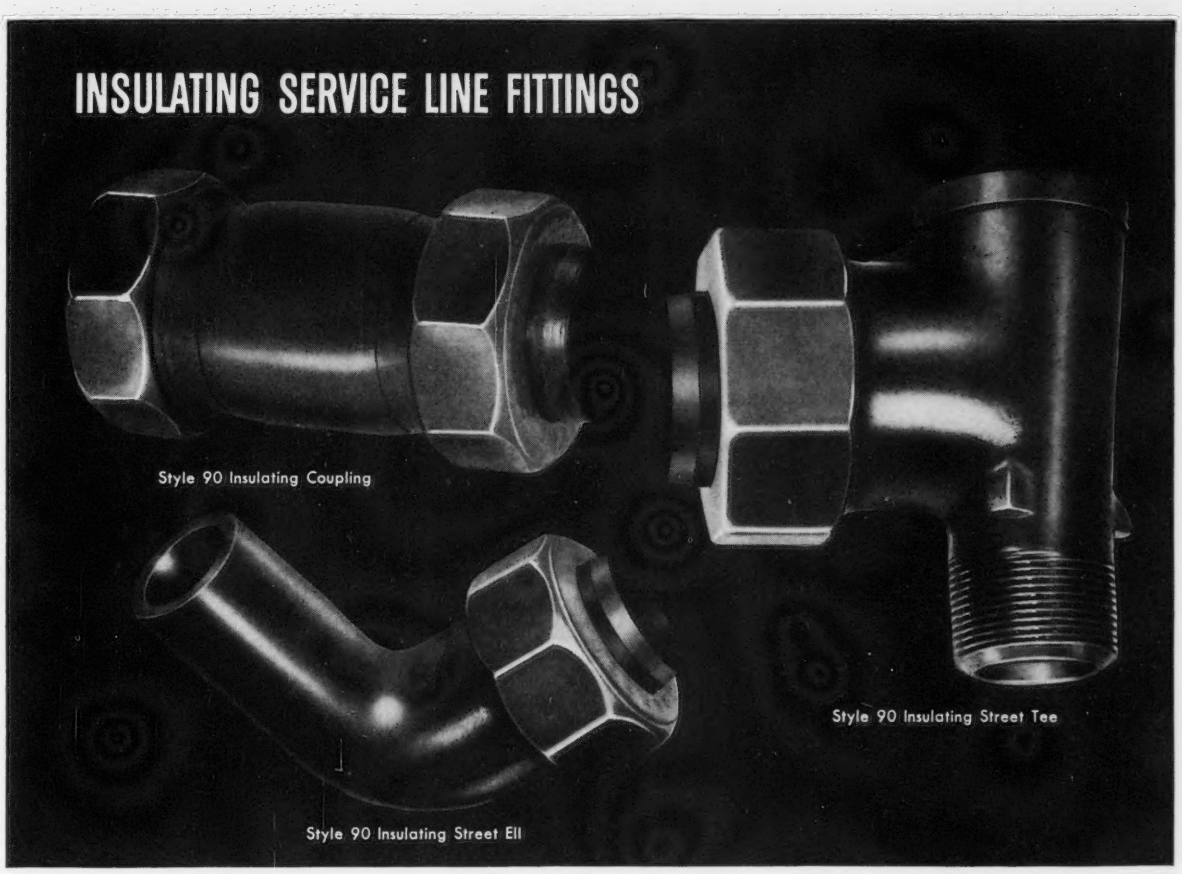
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(Continued From Page 86)

ber 1, 2 pm. The major subject at the T-2C meeting will be a report from the task group on current criteria. This task group was set up at a meeting in St. Louis last March.

Unit Committee T-2D Standardization of Procedures for Measuring Pipe Coating Leakage Conductance, Tuesday, October 1, 9 am. The committee will devote its time to the proposed standard method for measuring pipe coating leakage conductance on buried pipe lines.

Unit Committee T-2F on Internal Corrosion of Crude Oil Pipe Lines and Tanks, Wednesday, October 2, 9 am. The meeting will be devoted to a discussion of re-organization of the committee's activities. If interest in continuing the committee's work is not shown, it is possible termination proceedings will be started.

Unit Committee T-2J, Wrappers for Underground Pipe Line Coatings, Tuesday, October 1, 9 am. Suggested material specifications for asbestos felt, glass base outer wrap, rag felt and rock shield wrappers will be discussed. It is expected that these suggested specifications will be approved tentatively and made ready for letter ballot shortly.

Unit Committee T-2K Prefabricated Plastic Film for Pipe Line Coating, Tuesday, October 1, 2 pm. A suggested minimum required specification for prefabricated plastic film will be discussed. Reports from task groups on research and development and History and Results are expected also.

Unit Committee T-3G, Cathodic Protection, Wednesday, October 2, 2 pm. Progress reports from task groups on cathodic protection of ship hull bottoms and cathodic protection of process equipment will be heard. A report by task group T-3G-1 on cathodic protection of hull bottoms of ships will be discussed. This report was presented in tentative form at a meeting in San Antonio last October.

Unit Committee T-5C-1 Corrosion by Cooling Waters (South Central Region), Tuesday, October 1, 2 pm. T-5C-1 will hear reports from the work group on once-through and recirculating cooling water. It is expected progress of a test on stress corrosion cracking in cooling water equipment will be heard.

Unit Committee T-5E Stress Corro-

(Continued on Page 90)

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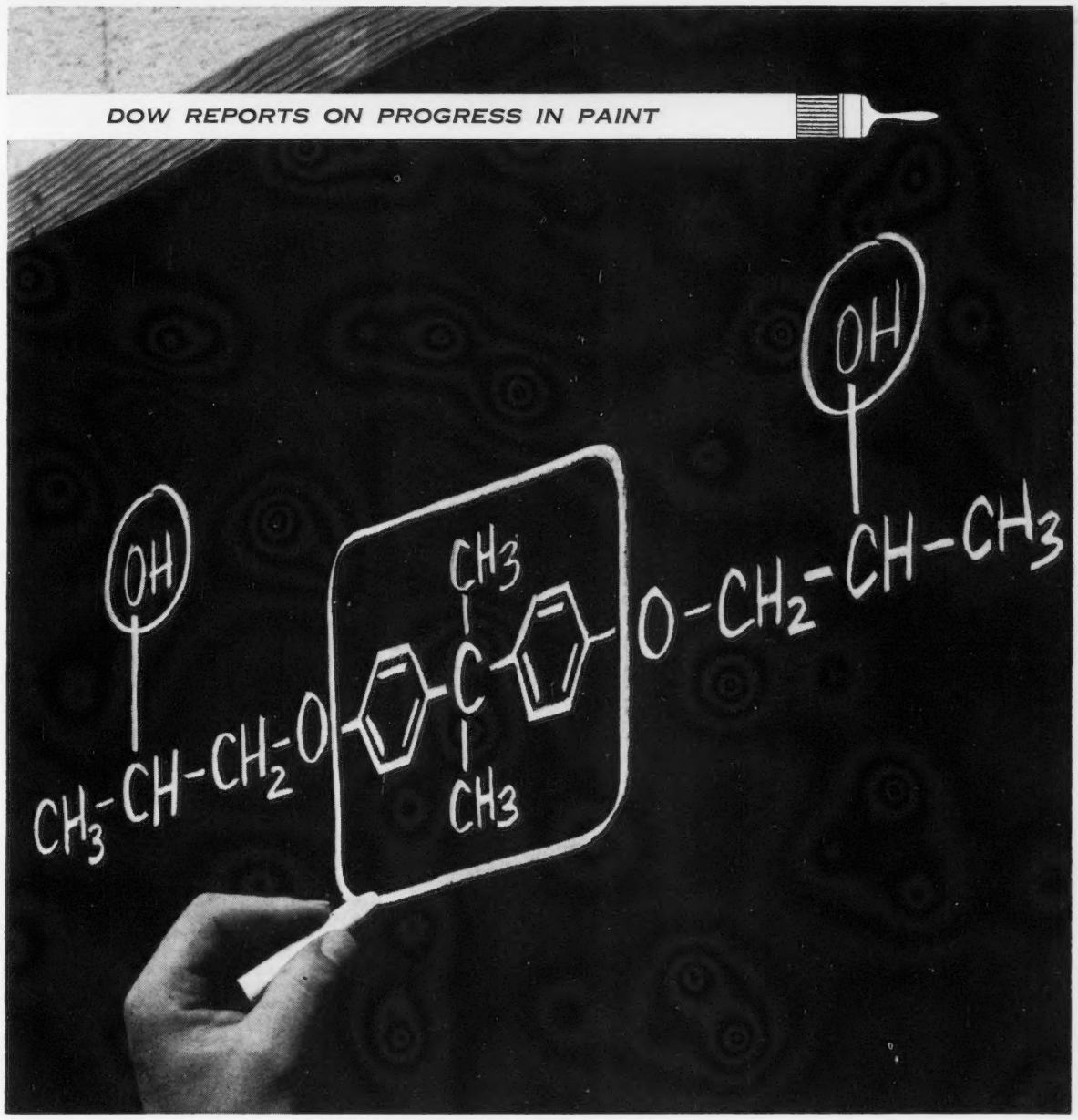

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(Continued From Page 88)

sion Cracking of Austenitic Stainless Steel, Wednesday, October 2, 9 am. A summary of a questionnaire on stress corrosion cracking of austenitic stainless steel will be discussed. T-5E is working jointly with an ASTM committee on the preparation of a report on stress corrosion cracking of austenitic stainless steels.

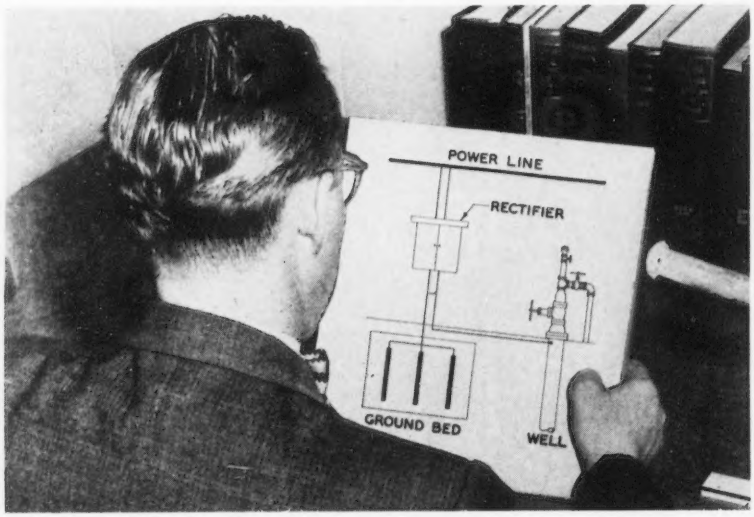
Unit Committee T-6B Protective Coatings for Resistance to Atmospheric Corrosion, Tuesday, October 1, 2 pm. T-6B will hear a report by a task group appointed at St. Louis to consider future work for the committee on protective coatings for resistance to atmospheric corrosion. The committee anticipates work on the following coatings: Linseed and other drying oils, ester gum oil, straight phenolic oil varnish, modified phenolic oil varnish, straight alkyd varnish, modified alkyd varnish, epoxy esters, epoxy (amine cured), chlorinated rubber, vinyls, metallic silicate, coal tar, and asphalt.

Unit Committee T-6D Industrial Maintenance Painting, Wednesday, October 2, 2 pm. T-6B will hear progress reports from task groups on economics of maintenance painting, standardization of scope for painting specifications and paint programs.

Unit Committee T-6E Protective Coatings in Petroleum Production, Wednesday, October 2, 9 am. Progress reports will be heard on protective coatings currently used in petroleum production.

Unit Committee T7-D South Central Region Corrosion Coordinating Committee, Wednesday, October 2, 2 pm. Organization of new corrosion coordinating committees and their activities will be discussed. A meeting of the Houston Coordinating Committee will be held at 9 am, Wednesday, October 2.

Unit Committee T-8 Refining Industry Corrosion. T-8 will hold two half-day meetings at 9 am on Tuesday, October 1 and beginning at 2 pm on Wednesday, October 2. High temperature hydrogen sulfide corrosion at high temperatures and pressures will be discussed. Other discussions will be on subjects such as naphthenic acid corrosion and acid cleaning. It is expected progress will be made with further organization of the committee formed at St. Louis.



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Dillon Heads Chemical Industry Committee

C. P. Dillon, Union Carbide Chemicals Co., Division of Union Carbide Corp., is the newly elected chairman of Technical Unit Committee T-5A on Chemical Manufacturing Industry. Mr. Dillon has been active for some years in NACE and has presented papers at regional conferences, at short courses on corrosion and at section meetings. Subjects for his technical papers include chemical process, cooling water and tanker corrosion.

Dillon

In addition to being chairman of T-5A he is chairman of T-5C-1 on Corrosion by Cooling Waters (South Central Region) and a member of committees T-3H on Tanker Corrosion; T-4E on Corrosion by Domestic Waters; T-5A-3 on Acetic Acid; and T-5D on Plastic Materials of Construction. He is the author of two articles recently published in CORROSIONS "Countermeasures for Control of Interal Corrosion of a Tanker Ship," and "Corrosion of Type 347 Stainless Steel and 1100 Aluminum in Strong Nitric and Mixed Nitric-Sulfuric Acids."

Since 1950 he has been a group leader of the corrosion and metallurgical group at the Texas City plant of Union Carbide Chemicals Co. He has been a member of NACE since 1947 and is a member of ACS and ASM also.

Chicago Meeting Set

Unit Committee T-7B North Central Region Corrosion Coordinating Committee will meet during the North Central Region sessions to be held at the Sherman Hotel in Chicago, October 2-4. Paul C. Hoy, Dayton Power & Light Company, Dayton, Ohio, chairman of T-7B will officiate. General functions of the NACE T-7 organization will be discussed.

Also to be discussed will be the relationship among existing electrolysis and corrosion coordinating committees in NACE T-7 organization. All members in North Central Region interested in corrosion coordinating committee work are urged to be present.

NACE NEWS

Golden Triangle Meeting Plant Tours Are Listed

Titles of Four More Papers on Program Are Revealed

Titles of four more of the 20 papers scheduled to be given during the Golden Triangle Northeast Region Meeting at Pittsburgh November 12-14 have been given. Nine plant tours also have been scheduled for those attending the sessions to be held at the Penn-Sheraton Hotel.

Additional information also was released as follows:

Hotel reservations should be mailed directly to the hotel.

A social hour will precede the Wednesday evening banquet at which the Westinghouse Quartet will perform.

A ladies' entertainment program has been completed.

Titles of Papers Given

The following titles of papers have been given in addition to those published in August on Page 98:

Corrosion Problems in the Chemical Industry—Use of Electrical Measurements to Predict Corrosion Rates, by R. B. Mears, U. S. Steel Corp.

New Metallurgical Developments Symposium—Some Newer Developments in Corrosion Resisting Steel and Steel Products, John Halbig, Armco Steel Corp., Middletown, Ohio. Properties of AM350, William Renshaw, Allegheny-Ludlum Steel Co., Brackenridge, Pa.

Instruments for Corrosion Symposium—Coating Evaluation, Harold C. O'Brien, Royston Laboratories, Inc., Blawnox, Pa.

Plant Tours Are Scheduled

The following plant tours have been scheduled for November 13:

Gulf Research and Development Company, Harmarville. Research, prospecting for and uses of oil are shown. Entomology laboratory for study of insecticides is included.

U. S. Steel Research Center, Monroeville, Pa.

Pittsburgh Plate Glass Co., Creighton. Manufacture of plate glass.

Continental Can Company, West Mifflin Borough. Container manufacture.

H. J. Heinz Co., Pittsburgh. Food processing and packing.

Aluminum Company of America, New Kensington, Pa.

Pittsburgh Coke & Chemical Co., Neville Island. Coal chemicals.

National Tube Co., McKeesport. Steel producing, tube piercing, finishing.

Fisher Scientific, Pittsburgh. Laboratory instruments and equipment.

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Nearly all of the technical material published in CORROSION appears nowhere else.

San Francisco Paper Industry and Oil And Gas Industry Symposia Are Filled

Two symposia to be presented at the March 17-21 NACE 14th Annual Conference and Exhibition at San Francisco are complete. The Paper Industry Symposium, with four papers and the Oil and Gas Production Symposium with five are among the first to be filled.

Material included in the Pulp and Paper Industry Symposium relates to organic materials, behavior of materials in "wet strength" solutions, problems involved in sulfur burning and acid making and a review of the recent work of the Technical Association of the Pulp and Paper Industry.

The number of booths available in the Civic Auditorium, San Francisco, Cal. for the March 18-20 NACE 14th Annual Exhibition has been increased from 108 to 120. This increase reflects confidence of responsible officials the show will be popular and demand for space heavy.

The 5-paper Oil and Gas Production symposium includes discussions of water flood operations, tubing, gas condensate wells, problems associated with dissolved gases and well casing.

Scheduled tentative are:

Paper Industry Symposium

Organic Resistant Materials in the Pulp and Paper Industry, R. P. Hill, Pulp Bleaching Corp., Seattle, Wash.

Review of Recent Corrosion Studies and Activities in the Technical Association of the Pulp and Paper Industry, S. J. Baisch, Thilmany Pulp and Paper, Kaukauna, Wis.

Materials, Behavior and Corrosion Characteristics in Paper Mill "Wet Strength" Solutions, I. S. Levinson, Ampco Metal, Inc., Milwaukee.

Corrosion Problems Encountered in Sulfur Burning and Acid Making in the Wood Pulp Industry, H. A. Lundberg, Seattle.

Oil and Gas Production Symposium

Corrosion of Steel by Varied Ratios of Dissolved Gases, J. Wade Watkins and G. W. Kinchloe, U. S. Petroleum Experiment Station, Bureau of Mines, Bartlesville, Okla.

Corrosion in Water Flood Operations, H. E. Greenwell and H. L. Bilhartz, Production Profits, Inc., Dallas.

Mechanical and Corrosion Resistant Properties of Oil Well Tubing, F. A. Prange, Phillips Petroleum Co., Bartlesville.

A Review of the Gas Condensate Well Corrosion Problem, D. R. Fincher, Tidewater Oil Co., Houston.

Cathodic Protection of Oil Well Casings in Ventura Field, Shell Oil Co., Los Angeles.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1957

Oct. 1-4—North Central Region, Chicago, Sherman Hotel. Exhibition.

Oct. 1-4—South Central Region, Oklahoma City, municipal auditorium. Exhibition.

Oct. 10-11—Southeast Region Meeting. Dinkler-Tutwiler Hotel, Birmingham, Ala.

Oct. 23-25—Western Region, Seventh Annual Conference, U. S. Grant Hotel, San Diego, Cal.

Oct. 23-25—7th Biennial Permian Basin Corrosion Tour. Midland-Odessa area, Texas.

Nov. 12-14—Northeast Region Fall meeting, Pittsburgh, Pa., Penn-Sheraton Hotel.

1958

Mar. 17-21—NACE Annual Conference, Civic Auditorium, San Francisco, California.

Oct. 5-8—Northeast Region, Somerset Hotel, Boston, Mass.

Oct. 15-17—North Central Region. Cincinnati, Ohio.

Oct. 20-24—South Central Region. New Orleans, Roosevelt Hotel.

1959

NACE Annual Conference, Sherman Hotel, Chicago, Illinois.

Oct. 12-15—South Central Region Meeting, Denver, Col.

SHORT COURSES 1958

October 22-23—Western Region. Corrosion Control Course (Evening Sessions), Hotel U. S. Grant, San Diego, Cal.

November 12-15—Miami Section Short Course, Key Biscayne, Fla.

December 9-13—Biennial Short Course on Cathodic Protection. University of Illinois and NACE.

Radio Program Scheduled On Western Region Meeting

A 30-minute radio program of a discussion including members of NACE will be broadcast October 26 in the San Diego area. The program is being prepared in connection with the October 23-25 Western Region meeting there.

It is expected also that a television interview of at least one person among those attending the meeting will be arranged.

NACE Fall Activity At Highest Level in 1957

Entertainment Committee Chairman Is Named

Harry Harner, Carpenter Steel Co., Alloy Tube Div., Pacific Northwest territory, San Francisco has accepted the post of entertainment committee chairman for the NACE 1958 Conference. He is a graduate of Staunton Military Academy and Washington and Lee University, Lexington, Va.

Harner

Budget for the conference was approved at the July 12 meeting of the NACE executive committee.

A descriptive brochure and contracts for space have been mailed to prospective exhibitors at the conference. Additional copies of this material can be obtained on request from Central Office NACE, 1061 M & M Bldg., Houston 2, Texas. After contracts have been executed and returned to NACE booth space will be allocated according to the formula developed over the past several years.

1959 South Central Meet Will Be Held in Denver

The 1959 meeting of South Central Region has been set for October 12-15 at Denver, Col. according to an announcement by Jack P. Barrett, Pan American Petroleum Corp., Tulsa, chairman of the region.

The 1958 meeting of the region will be held in New Orleans.

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Seven major corrosion coordinating committees have affiliated with NACE.

More Oklahoma City Exhibitors Are Listed

In addition to the nineteen firms listed as purchasers of space at the Oklahoma City exhibition of South Central Region in October the following have reserved space:

Agra Instrument Company, Tulsa, Okla.
S. E. Bosley Company, Tulsa, Okla.
Crutcher-Rolfs-Cummings, Inc., Houston, Texas
Engineering Test Services, Inc., Tulsa, Okla.
F. H. Maloney Company, Houston, Texas
Nocor Chemical Company, Odessa, Texas
Pittsburgh Coke & Chemical Company, Pittsburgh, Pa.
Pipeline Coating & Engineering Co. Inc. and Plicoflex, Inc., Los Angeles, Calif.
Protecto Wrap Company, Tulsa, Okla.
Rio Engineering Company, Houston, Texas
Royston Laboratories, Inc., Pittsburgh, Pa.
D. E. Stearns Company, Shreveport, La.

Field Exhibits Set Up For Permian Basin Tour

Field exhibits of oil field production and refinery equipment have been scheduled for the Seventh Biennial Permian Basin Corrosion Tour October 23-25. Tour headquarters will be in Odessa. Many of the exhibits of equipment will be same as have been inspected during previous tours, permitting evaluation of control measures used.

The $20 registration fee includes transportation by air conditioned buses to Goldsmith and South Cowden Fields, waterflood projects in the Monahans-Kermit area, McElroy Field near Crane, Vealmoor area north of Big Spring, Howard-Glasscock Pool in the Forsan vicinity, Midland Farms and others. E. O. Kemper, Box 4132 Odessa, will accept advance registrations.

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More than 10,000 NACE Corrosion Abstract Cards have been published so far.

10 Multiple-Day Events to Be Held Before Year's End

The pattern of activity in the National Association of Corrosion Engineers has changed so rapidly in the past several years that the fall of the year has more events than the spring. Seven multiple day meetings and three short courses will be held before the end of the year. Of these meetings, five are regional meetings and two are section-sponsored activities. One short course is region-sponsored and two others are section activities.

Titles of 58 papers to be given at three regional meetings have been published and it is likely that 20 or more will be given at sessions whose programs have not been made public. The subject matter ranges from coatings to underground corrosion.

Technical committee meetings have been planned for at least two of the regional meetings. The number of NACE sessions in the fall and their wide geographical dispersion leads to the assumption that most technical committees may find it practical to meet twice yearly, instead of only once at the national NACE conference as at present.

All of this activity is taking place while arrangements for the annual conference at San Francisco next March are rapidly being completed. Principal emphasis during the remainder of the year probably will be on the exhibition, although some symposia need additional manuscripts.

Scheduled this fall are:

Oct. 1-4—South Central Region Meeting, Municipal Auditorium, Oklahoma City. Six symposia, 24 papers, principally about the oil and gas industry. Complete roundup of information published in August beginning on Page 90. Exhibition.

Oct. 1-4—North Central Region Meeting, Sherman Hotel, Chicago. Eight symposia and educational lectures, 35 papers, chemical industry, utilities, underground. Roundup of information in this issue.

Oct. 10-11—Southeast Region Meeting, Dinkler-Tutwiler Hotel, Birmingham, Ala. Twelve papers, some about corrosion problems peculiar to the Southeast.

Oct. 23-25—Western Region Meeting. U. S. Grant Hotel, San Diego, Cal.

Oct. 23-25—7th Biennial Permian Basin Corrosion Tour, Midland-Odessa area.

Oct. 22-23—Corrosion Control Short Course, Western Region, concurrent with regional meeting.

Nov. 12-14—Northeast Region Meeting, Penn-Sheraton Hotel, Pittsburgh. Six symposia, 20 papers on chemical industry, coatings, instruments, metallurgy.

Nov. 13-16—Miami Section Meeting, Key Biscayne, Fla. Technical papers to be given. Concurrent corrosion control short course.

Dec. 9-13—Biennial Short Course on Cathodic Protection, University of Illinois. Roundup of information published in June, Page 98.

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It may be your lucky day!
If, so, we'll be glad to pay.
Later, you'll be glad to say ...
'I now know of Gulf-State's way
of enamel resistance to
Impact ... Shock ... Stress ... and Play'..."

We're mighty proud of impact and shock resistance we've been able to formulate into Gulf-States pipe enamels and primers. So proud, in fact, that we're paying $100.00 a day to the person making the closest estimate as to the number of times a GS sample has been struck by a one-pound ball in a standard test machine.

VISIT BOOTH NUMBER 27 AT THE OKLAHOMA CITY EXHIBIT
—YOUR MATHEMATICAL CALCULATIONS MAY PAY OFF!

Corrosion Problems of Southeastern U. S. To Be Covered in Birmingham Meeting

Corrosion problems in the Southeast that may not be common in other parts of the United States will be discussed at a meeting in Birmingham, Ala. during October. Southeast Region has scheduled sessions October 10-11 at the Dinkler-Tutwiler Hotel where these problems will be considered. A report outlining some corrosion problems peculiar to the South will be presented.

Some ten other papers are to be given, including "Breakdown Value of Tests Made on Coatings Used in the South," "Corrosion Encountered on Several Telephone Systems" and "New Interpretations in Cathodic Protection."

Arthur B. Smith, Amercoat Corp., Jacksonville, Fla., Southeast Region chairman is in charge of the meeting. Joseph Frink, Florida Power & Light Co., Miami, regional vice-chairman is in charge of the program assisted by John B. Paisley, Jr., American Telephone and Telegraph Co., Birmingham section program chairman.

Genesee Valley Section Elects Levy Chairman

At the final meeting of the season, Genesee Valley Section elected the following new officers to serve for 1957-1958: A. F. Levy, The Pfaudler Company, chairman; Paul T. Lahr, Goulds Pumps, Inc., vice-chairman; and John V. Adkin, Rochester Gas & Electric Corp., secretary-treasurer.

ERRATA

The schedule of awards to Young Authors listed on Page 82 of the 1957 NACE Yearbook should be as follows:

1952—B. W. Dunnington and F. H. Beck.

1953—P. M. Aziz.

1954—Robert A. Lad.

1955—E. L. Simons.

1956—Thor N. Rhodin.

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The following biography should be substituted for that appearing on Page 123 in the June, 1957 issue of CORROSION:

H. O. TEEPLE
International Nickel Co., Inc.
New York, N. Y.

I graduated from the University of Michigan in 1937 (B.Sc. Ch.E.) and joined Westvaco Chemical Division of Food Machinery and Chemical Corp., at South Charleston, West Virginia, where I worked on materials specification and plant corrosion problems. In 1944, I joined the International Nickel Co., Inc., as a member of the Corrosion Engineering Section. Among those fields in which I have specialized is the Pulp and Paper Industry. I am a member of NACE; the Technical Association of the Pulp and Paper Industry; the Canadian Pulp and Paper Association (Technical Section); the Electrochemical Society; and the American Chemical Society.

At the present time (through 1958) I am General Secretary of the TAPPI Engineering Division and a member of the TAPPI Corrosion Committee.

Anaerobic Corrosion Is Topic at San Antonio

Anaerobic corrosion was discussed at the July 16 meeting of Alamo Section by J. W. Gibson of D. W. Haering & Co., Inc. Mr. Gibson pointed out this type of corrosion occurs widely in oilfield and oil refinery structures on equipment ranging from pipelines to heat exchangers. Some workers consider corrosion resulting from action of sulfate reducing bacteria to be second only to stray current electrolysis as a cause of pipeline failures.

Dictaphone Equipment Is Given by S. Central Region

Dictaphone recording equipment valued at more than a thousand dollars has been given to NACE for national organization use by the South Central Region. The equipment was purchased in September 1955 by South Central Region.

The unit includes six microphones with an electronic mixer box to enable the operator to switch and control recording volumes from each of the individual microphones. It can record continuously without interruption, and can be connected to public address systems.

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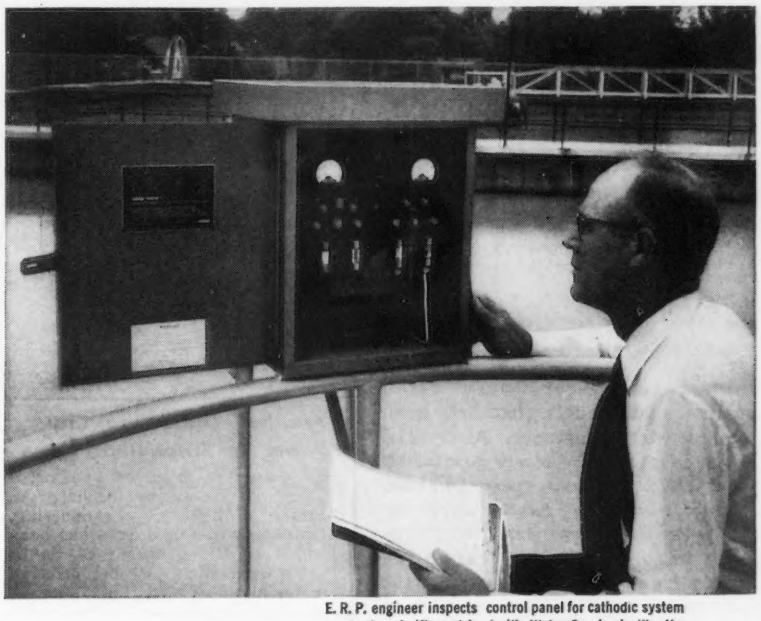
Subscribers to CORROSION are found in over 40 foreign countries.



E. R. P. engineer inspects control panel for cathodic system protecting clarifiers at Louisville Water Co., Louisville, Ky.

E. R. P. CATHODIC PROTECTION
stops corrosion at lowest overall cost

Electro Rust-Proofing Cathodic Protection is used by the Louisville Water Company to prevent corrosion of metal underwater equipment in the raw water clarifiers.

Cathodic protection is the one method able to reduce corrosion of underwater metal virtually to zero. Electro Rust-Proofing Cathodic Systems are designed to provide the sole protection of submerged structures or to supplement a good protective coating. When used in conjunction with coatings, cathodic protection eliminates corrosion due to holidays or inherent breaks in coatings such as can occur at many inaccessible structural joints.

Electro Rust-Proofing designs every cathodic system individually so that maximum protection is given for the lowest operating cost. For information about E.R.P. Cathodic Protection, write for Bulletin #E-39.

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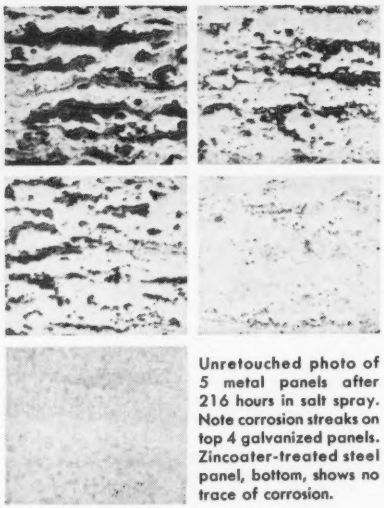

Unretouched photo of 5 metal panels after 216 hours in salt spray. Note corrosion streaks on top 4 galvanized panels. Zincoater-treated steel panel, bottom, shows no trace of corrosion.

Salt Spray Test Proves ZINCOATER Resists Corrosion *Better than Galvanizing*

New galvanized iron panels from four manufacturers were tested against steel panels that had been treated with Zincoater. After 216 hours in salt spray, *every galvanized panel showed some trace of rust. Zincoater-protected panels were entirely free from corrosion.*

Its high corrosion resistance makes Zincoater the ideal primer for galvanized and steel surfaces such as signs, buildings, water tanks, ducts, dams and railroad and truck bodies. Zincoater's special formula bonds tightly, seals out corrosion-causing moisture and increases durability of finish paint coats.

Before you paint galvanizing or submerged steel, prime with Tropical Zincoater!

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Heavy-Duty Maintenance Paints Since 1883

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Southwestern Ohio Section Includes Indiana Towns

Southwestern Ohio Section has expanded its boundaries to include a portion of Southeast Indiana. Members residing in the following Indiana cities are now members of the Southwestern Ohio Section: Bluffton, Fort Wayne, Indianapolis, Kokomo, Lebanon, Mt. Vernon, Terre Haute and Zionsville. The expansion adds 31 members to the section's membership. The tentative 1957-1958 program for the Southwestern Ohio Section is as follows:

September 23, Columbus, Ohio—Sponsored by North American Aviation Company and Battelle Memorial Institute, subject: "Combined Inspection Tour and Meeting on Atomic Corrosion."

October 29, Dayton, Ohio.

November 26, Cincinnati—Sponsored by National Distilleries, subject: "Chemical Corrosion."

December 17, Cincinnati, Ohio—Christmas Party.

January 28, 1958, Cincinnati—Sponsored by Cincinnati Gas & Electric Company, subject: "Underground Corrosion."

February 25, Zionsville, Indiana—Sponsored by Dresser Industries, subject: "Corrosion in Meters and Blowers."

March 25, Hamilton, Ohio—Sponsored by Champion Paper Company, subject: "Paper Industry Corrosion."

April 29, Dayton, Ohio—Sponsored by Dayton Power & Light Company.

May 27, Cincinnati—Subject: "Refinery Corrosion and Oil Additives."

Another Oklahoma City Paper Is Scheduled

"Engineering Colleges vs Corrosion," a paper by Richard V. James, College of Engineering, University of Oklahoma, has been added to the program of South Central Region's October meeting in Oklahoma City.

Shreveport Barbecue Set

Shreveport Section will hold its annual barbecue at Lake Bistineau September 6. Section friends are invited.

Frank Moran, Moran Tank Co., presented a motion picture "Zinc Control Corrosion" at the section's August 6 meeting attended by 35.

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SECTION CALENDAR

Sept.
23 Tulsa Section.
24 East Texas Section.
No Date Metropolitan New York Section.
No Date North Texas Section.

Oct.
9 Tidewater Section.
29 Philadelphia Section. Panel, "Materials of Construction."

Ladies' Night Sept. 17 Opens Chicago Schedule

Ladies' night September 17 opens a 7-meeting schedule for Chicago Section. Final meeting is to be held during May, 1958.

The schedule follows:

Nov. 19—Field Evaluation of Pipeline Coatings, M. E. Parker, Consulting Engineer, Houston.

Jan. 21—Use of Inhibitors in the Oil and Chemical Industry.

Feb. 18—Properties and Uses of Epoxy Resins, H. W. Howard, Shell Chemical Corp.

Mar. 18—Use and Application of Aluminum and Aluminum Alloys in the Oil and Chemical Industry, R. W. Flournoy, Reynolds Metals Co.

April 15—Metallurgical Aspects of Corrosion.

May 25—Laboratory Development of Protective Coatings, H. C. O'Brien, Royston Laboratories; Field Evaluation of Industrial Maintenance Coatings, Kenneth Tator, K. Tator Associates.

WILLIAM J. ANDERSON

William J. Anderson, Dalzell Electric Welding Co., Ltd., Bellshill, Scotland was killed in a motor accident May 25. Mr. Anderson, a member of NACE, was managing director of his company.

Polyethylene Properties Topic of SPE Session

Eight technical papers on the properties and uses of polyethylenes will be given at an October 17 meeting in Cleveland. The program, sponsored by the Society of Plastics Engineers, Inc., will be held at Hotel Carter.

Morning session will consider history, low density, medium density and linear polyethylene. The afternoon papers will be on blow molding, injection molding, pipe extrusion and film.

Advanced registrations may be sent to E. J. Haskins, Zenith Plastics Co., 3901 Superior, Cleveland 14, Ohio. Fee is $9, including luncheon and preprints.

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The NACE Abstract Filing System is used by many organizations.

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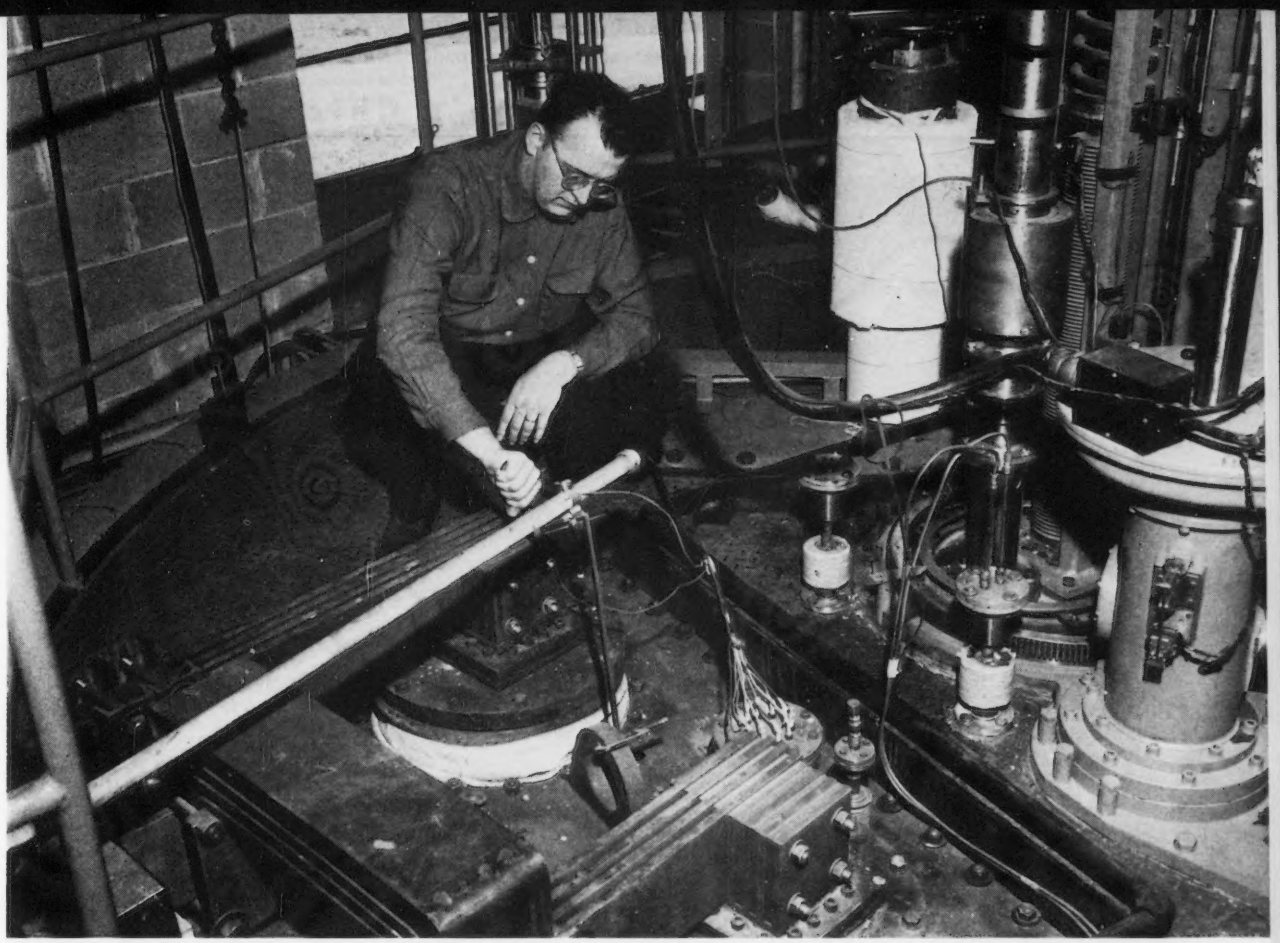
Nearly all of the technical material published in CORROSION appears nowhere else.



F. A. Smith, one of the authors on the Chicago program is shown in the photograph above at work on an atomic reactor. Mr. Smith has worked on atomic energy problems since 1943. He will discuss liquid metal corrosion on the afternoon of October 3.

Chicago Program Will Consider Major Problems

A comprehensive 35-paper technical program arranged for the October 2-4 meeting in Chicago of North Central Region covers a wide variety of information in several major industrial fields. The sessions, to be held at the Sherman Hotel, also will include a demonstration and a discussion.

This will be the second meeting of North Central Region, which prior to 1956 did not hold technical sessions. The variety of information included in the technical papers scheduled indicates growing interest in the region in corrosion control problems.

The program also includes a well-rounded social program and a color motion picture on Russia during the annual banquet October 2 narrated by National Broadcasting Company commentator Alex Dreier.

A novel feature of the meeting is an invitation to students of 35 engineering colleges to attend the sessions. This action was taken in belief engineering students may want to consider seriously the merit of specializing in corrosion control. The meeting will give them an opportunity to learn something of progress being made in corrosion control and discuss it with active corrosion workers.

Social Events Planned

Social events include a Fellowship Hour to be held Wednesday, October 2; a ladies' program consisting of three tours and several luncheons and the annual banquet October 3.

Registration begins Tuesday and continues through Thursday. Admission to

(Continued on Page 96)

SCHEDULE OF CHICAGO SYMPOSIA IS GIVEN

October 2

Morning: Protective Coatings, Part 1; Water Treating.

Afternoon: Protective Coatings, Part 2; Pipe Line Corrosion.

October 3

Morning: Cathodic Protection, Transportation.

Afternoon: Chemical Processing Industry, Cathodic Protection Round Table Discussion.

October 4

Morning: Refinery Corrosion, Public Utilities.

Afternoon: Educational Lectures.

BIOGRAPHIES

E. B. BACKENSTO—Supervising Engineer in charge of corrosion group with Socony Mobil Oil Company, Inc. at its Research and Development Laboratory, Paulsboro, New Jersey. He has been with Socony Mobil since receiving a BS in chemical engineering from Lehigh University in 1943. He has been a group leader in charge of light products refining and treating, as well as reforming catalyst development. He is a member of AIChE, ACS, NACE and API Subcommittee on Corrosion and is vice-chairman of NACE's Refining Industry Corrosion Committee T-8.

J. C. BARBER—Assistant Chief, Plant Chemical Control Branch, Tennessee Valley Authority, Office of Chemical Engineering. He directs the technical investigative program in the authority's large-scale chemical plants, specializing in production and processing of elemental phosphorus to produce phosphate fertilizers. A graduate of Georgia Institute of Technology, with a BS in ChE (1938), he has worked for TVA since graduation and is a member of ACS and AIChE.

RICHARD BENES—Corrosion engineer, Technical Division, Corn Products Refining Co., Argo, Ill. He attended University of Illinois where he received a BS in chemical engineering in 1953. He is now occupied with corrosion testing and

(Continued on Page 96)

Authors—North Central Region's Chicago Meeting

Backensto

Barber

Benes

Bloom

Bogumill

Brooks

Dieman

Draley

Dravnieks

Drew

Francis

Gaynor

Geyer

Gleekman

Hamel

Haygood

Holsinger

Kiernan

BIOGRAPHIES

(Continued From Page 95)

evaluation methods and is a member of AIChE and NACE.

MORTIMER C. BLOOM—Corrosion Consultant, Metallurgy Division, U. S. Naval Research Laboratory, Washington, D. C. He has a BS in chemical engineering from Massachusetts Institute of Technology (1922). After six years in the paper and petroleum industries he returned to MIT to earn a PhD in physical chemistry and work as a research associate in crystal chemistry and electrochemistry. He became a consultant to the NRL in 1949 after several years as a private consultant.

THOMAS R. BOGUMILL—Assistant General Distribution Superintendent of The Peoples Gas Light and Coke Company of Chicago. During his 33 years with the company he has worked in several departments, but has devoted most of his time to distribution maintenance and new construction. He has a BS in chemical engineering, University of Wisconsin (1923) and recently completed a session in advanced management at Harvard.

W. R. BROOKS—He has been with Binks Manufacturing Company for the past 13 years, principally occupied in testing equipment and trouble shooting in the field as well as conducting the company's spray painting school. He is a graduate of St. Ambrose College.

EDGAR A. DIEMAN—A research chemist at Standard Oil Co. (Indiana) Whiting, Ind., he has been active in research on rust preventives since joining Standard in 1947. He previously was technical supervisor at Nox Rust Chemical Corp., Chicago and chief plant chemist, Cities Service Corp., East Chicago, Ind. He has a BS from Carroll College (1931) and an MS in organic chemistry, University of Wisconsin (1933). He is a member of ACS.

JOSEY E. DRALEY—He has been active in corrosion research since he joined the staff of the Metallurgical Laboratory, University of Chicago in 1942 and in the atomic energy field almost continuously with the Kellex Corp., Oak Ridge National Laboratory and Argonne National Laboratory. He has worked largely on corrosion mechanisms of aluminum, uranium and zirconium and their alloys in pure water and dilute solutions. He is author of numerous project publications and active as a speaker at meetings of NACE and The Electrochemical Society. He currently is chairman of the Corrosion Division of The Electrochemical Society and Corrosion Division Editor of its journal.

ANDREW DRAVNIEKS—Head of the Corrosion Section, Material Division, Engineering Research Dept., Standard Oil Company (Indiana). He attended University of Latvia and Marburg University, Germany and received a PhD in 1949 from Illinois Institute of Technology. He has done work in paint technology, polarography, surface chemistry and in the kinetics of corrosion processes.

R. D. DREW—Assistant Supervisor of Process Development Section, Research and Development Laboratory, Socony Mobil Oil Company, Inc., Paulsboro, New Jersey. He has been with Socony Mobil since receiving his bachelor's degree in chemical engineering at Yale University in 1939. A member of AIChE, ACS and Sigma Xi, his work with Socony-Mobil has been mainly in development of continuous catalytic cracking and reforming processes and catalysts.

HOWARD T. FRANCIS—Supervisor of the Electrochemistry Section of the Armour Research Foundation, he received a BS in chemistry from Mount Union College in 1938 and a

(Continued on Page 98)

Chicago Program—

(Continued From Page 95)

the Fellowship Hour is by registration badge.

Detailed information on these events was published in CORROSION, August, 1957 on Page 88.

Roundup of Program

A roundup of the technical program, including available abstracts, latest information on titles and biographies of authors is included in this issue. A survey of the technical papers shows data pertaining to the chemical processing, petroleum refining, public utility and transportation industries will be presented.

Other papers pertain to cathodic protection, coatings and pipe line corrosion. Five papers are scheduled on various aspects of water treating as are three lectures on some of the fundamental problems associated with corrosion processes.

A soil-box demonstration on the galvanic corrosion of metals underground and on the effect of cathodic protection also will be given. This graphic presentation helps clarify some of the principles applicable to electrical protection of metals.

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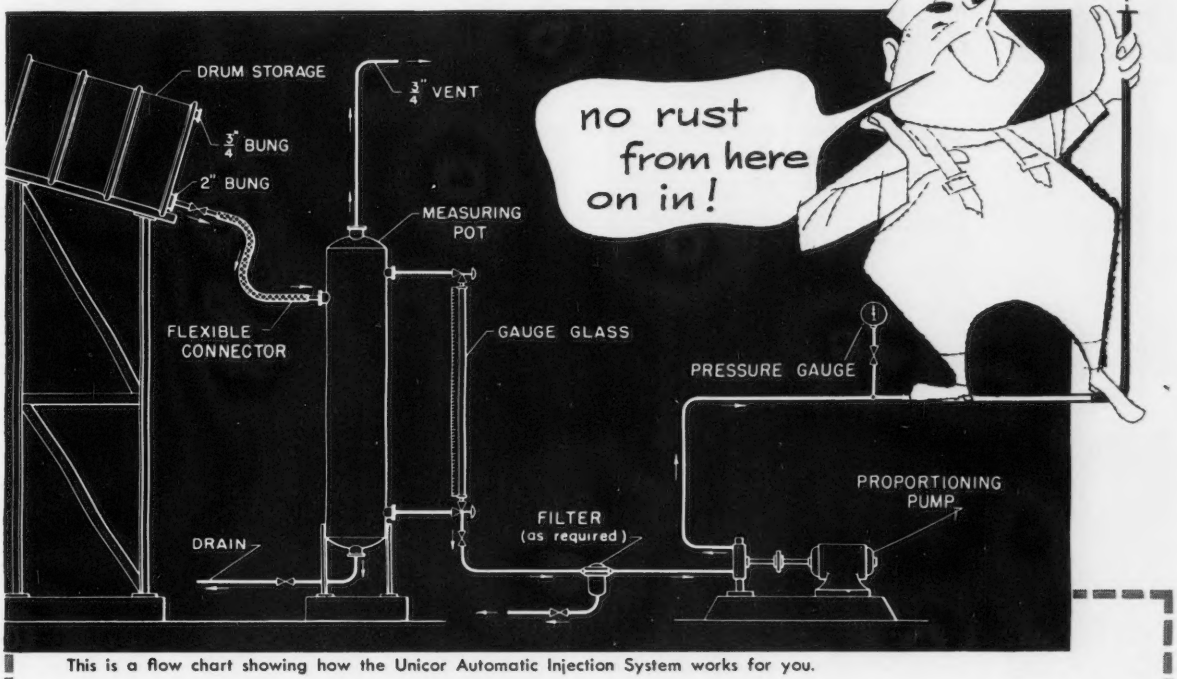

This is a flow chart showing how the Unicor Automatic Injection System works for you.

Question: *How can we eliminate serious filter fouling in blending tank caused by formation of highly peptized iron rust during processing?*

Answer: UOP's Unicor corrosion inhibitor solved this problem for a large southern refiner not long ago. Before Unicor application, a yard-square panel of the tank interior was sandblasted down to bare metal to test Unicor's effectiveness. After several months of injection in the rundown line, this test panel was completely free of corrosion, the remainder of tank interior was free of filter-fouling rust.

Question: *Is addition of water necessary?*

Answer: No. Unicor is oil soluble. It also prevents rusting caused by unavoidably entrained or dissolved moisture.

Question: *Should Unicor be injected only in the rundown line?*

Answer: Unicor can be injected at any point in your process. The refiner mentioned above shifted the Unicor injection point from the rundown line to his crude fractionator overhead and in catalytic gasoline from the main column overhead receiver. This extended Unicor's protection to heat exchangers, condensers and towers between new injection points and blending tank. Unicor inhibits corrosion in all your processing equipment from the point of injection on... even to consumer equipment.

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Authors—North Central Region's Chicago Meeting

Koenecke

Kretschmer

Kroon

Larson

Marsh

McDougall

Meyer

Miller

Minford

Murphy

Peabody

Peterson

Pierson

Ploederl

Prior

Rose

Schaschl

Segool

BIOGRAPHIES

(Continued From Page 96)

PhD from Pennsylvania State College in 1942 when he joined the foundation. His fields of interest include corrosion, electrodeposition and batteries with major emphasis on new solutions of electrochemical problems.

J. WILLIAM GAYNOR—Group Leader in charge of research on industrial lubricants, he joined research department Standard Oil Co. (Indiana) Whiting, Ind. in 1937. He has a BA from Yankton College and an MS in organic chemistry from University of Michigan. He is a member of ACS and ASLE.

JOHN H. GEYER—Manager, Development Department, American Chemical Paint Co., Ambler, Pa., he is a 1949 graduate of Lehigh University with a BS in metallurgical engineering. He worked for Aluminum Company of America before joining American Chemical Paint Co. in 1951. He was made manager of its development department in 1953.

L. W. GLEEKMAN—Supervisor of Materials Engineering, Research and Engineering Division, Wyandotte Chemicals Corp., Wyandotte, Mich. He has a B ChE from Cooper Union Institute, New York and an MS and PhD in chemical engineering from State University of Iowa. He has been employed as a metallurgist for Sam Tour & Co., New York prior to military service in Chemical Warfare Service during World War II; was assistant professor of chemical engineering at University of Delaware for four years, and for a year was research engineer with Sherritt Gordon Mines in Ottawa, Canada before joining Wyandotte Chemicals in 1953. He also is teaching evening extension courses in metallurgy at Detroit Institute of Technology and corrosion at Wayne University. He is a member of NACE, ASM, AIChE and ASEE. He is chairman of T-5a-4 on Corrosion in the Chlorine Manufacturing industry.

F. B. HAMEL—Currently materials and corrosion specialist in the Manufacturing Department, General Engineering Division, Standard Oil Company of Ohio, Cleveland. Previously he had six years experience in shop inspection, project and planning work. A 1948 graduate of Ohio State University with a BS in mechanical engineering, he spent 4½ years in the army during World War II.

ASHBY J. HAYGOOD—An engineer on the Aluminum Company of America's sales development division staff, he is a specialist on application of aluminum in the appliance field. He has a BS in mechanical engineering, Alabama Polytechnic Institute (1950), joining Alcoa on graduation. From June 1951 to April 1953 he was in the Army Corps of Engineers in Korea. He contributed to development of an all-aluminum steam iron and to components of other appliances. He is an associate member of American Society of Heating and Air Conditioning Engineers.

FRED J. HOLSINGER—He is a corrosion metallurgist with Research and Development Dept., Corn Products Refining Co. He has a BS in chemical engineering, University of Notre Dame (1941), joining his present employer in 1946 after duty with the navy in World War II. He has been active in corrosion work since 1932.

FRANK J. KIERNAN—Technical Supervisor of Protective Coatings Section, Barrett Division, Allied Chemical & Dye Corp., New York City. He has been successively assistant project engineer, Wyandotte Chemical Corp. (1948-50); research and development engineer, U. S. Stoneware Co. (1952-55). He has a BS in chemical engineering, University of Detroit and associate degrees in ceramics and petroleum engineering, with graduate work in nuclear engineering. Among other organizations he is a member of NACE, AGA, AWWA, API, ACS.

DONALD F. KOENECKE—He has spent 11 years developing new coating resins for Esso Research and Engineering Co., Linden, N. J. He has a BA from St. Olaf College and an MS in chemistry from North Dakota State College.

W. J. KRETSCHMER—Senior Corrosion Engineer, Gas Engineering Dept., Columbia Gas System Service Corp., Columbus, Ohio. Almost all of his time since 1939 with the Service company has been occupied with corrosion control of pipelines, cables and other utility property. He attended the electrical engineering department of Ohio State University College of Engineering, is a member of several NACE technical committees, of the AGA corrosion committee and past chairman of the AGA subcommittee on underground corrosion.

THURSTON E. LARSON—He is responsible for the laboratory which makes analyses for minerals in water and does research on water treatment methods, particularly with respect to corrosion, softening and analytical methods. Frequently an author and co-author of technical papers on water, he is active in NACE technical committee work, and a member of ACS, AWWA, AAAS and other organizations. He is head of the Illinois State Water Survey's chemistry section and is its assistant chief. He has a BS and PhD from University of Illinois, 1932 and 1937.

GLENN A. MARSH—Pure Oil Company, Research and Development Laboratories, Crystal Lake, Illinois. He received a BS in chemistry from Illinois Institute of Technology in 1945 and an MS in chemistry from Northwestern University in 1946. He began his employment with Pure Oil Company in 1948 and is presently engaged in corrosion research.

ROBERT I. McDOUGALL—Now evaluating pipeline coatings in the Chemicals Research Division, Esso Research and Engineering Co.,

(Continued on Page 100)

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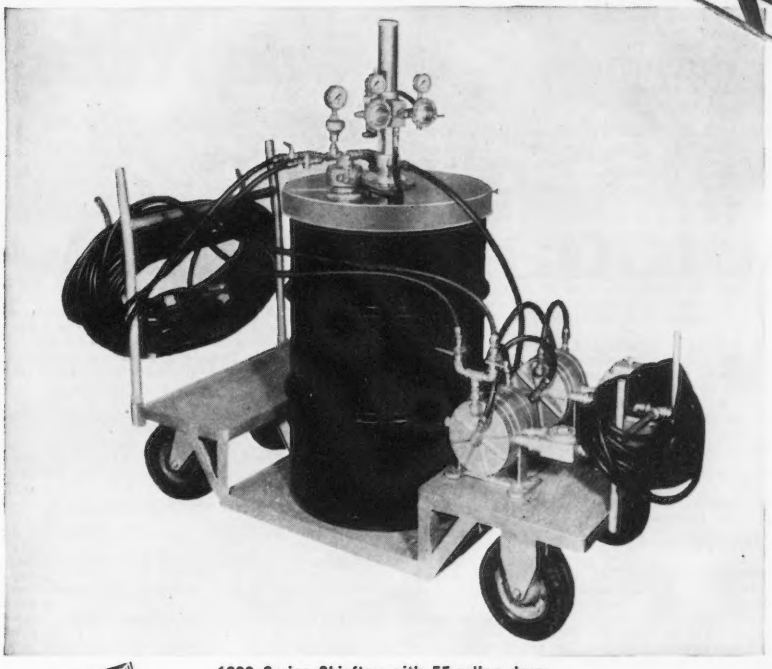

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Authors—North Central Region's Chicago Meeting

Shields

Sjoberg

Skold

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Stern

Stutz

Sudrabin

Waldron

Walston

Watkins

BIOGRAPHIES

(Continued From Page 98)

Linden, N. J., he joined Esso after receiving a PhD in physical chemistry from Carnegie Institute of Technology (1956). He has an AB in chemistry from Indiana University (1951) and is a member of NACE, among other organizations.

ROBERT H. MEYER—Project engineer for Sinclair Pipe Line Company, Independence, Kansas, he is now in charge of liaison with Sinclair Research Laboratories, Inc. on pipe line research and development projects. He has a BS in chemical engineering (1949) Kansas State College and was with Phillips Petroleum Corp. for a year. For two years he was with the Oil Movement Dept. of Sinclair and since then a member of the engineering department. He is a member of NACE.

JAMES DEAN MINFORD—Research Engineer in the New Kensington, Pa. research laboratories of Aluminum Company of America, he has a BS in chemistry, Carnegie Institute of Technology (1943) and MS and PhD in physiological psychology and bio-organic chemistry in 1948 and 1951. Since 1953 with Alcoa he has worked on inhibitors for aqueous and chemical environments and on studies of reaction mechanisms involving aluminum and organic chemicals.

HUGH R. MURPHY—Senior Engineer, Distribution Department, South District, The People's Gas Light and Coke Company, Chicago, he has a BS, University of Illinois (1949) received after serving five years in the Air Force. When he joined his present employer in 1949 he assisted in the design, inspection and installation of high pressure gas transmission lines for Chicago District Pipeline Company. During the past six years he has worked in both the service and distribution departments of the company's South District.

DAVID C. PALM—General Superintendent of Pipelines for Natural Gas Pipeline Co. of America and affiliated companies. After graduation from Stanford University, he began work with Cities Service Company in engineering and has been with his present employer since it was organized. His experience covers construction of oil and gas refining, processing, transmission and measuring facilities. He is active on several committees of AGA related to pipeline transmission and maintenance.

A. W. PEABODY—Electrical engineer for Ebasco Services Inc. since his graduation in 1937 from University of Maine, except for military service. Since 1939 he has been occupied with design, survey, construction supervision and adjustment of corrosion control installations for cathodic protection systems on pipe, cable and generating station facilities. He is frequently a lecturer at NACE meetings and short courses.

N. H. PETERSON—A member of the Consultant Staff (Corrosion) in the Metallurgy Division, U. S. Naval Research Laboratory, Washington, D. C. He has a BS in chemistry, Clemson College (1944) and MA in analytical chemistry from University of North Carolina (1951). He has been an instructor in the Clemson College chemistry department and associated with the Naval Ordnance Laboratory, White Oak, Md. in the evolution of electrochemical ordnance devices. He has worked at NRL since 1954 with a group occupied with cathodic protection of naval vessels.

RUSSELL E. PIERSON—A corrosion engineer in the Technical Division of Corn Products Refining Co., Argo, Ill., he has a BS in chemical engineering, State University of Iowa (1952). Before joining Corn Products Refining Co. in 1954 he was in the U. S. Army Chemical Corps. He is a member of AIChE and NACE.

F. J. PLOEDERL—In charge of coating research and development with Wisconsin Protective Coatings Company, Green Bay, Wis. He has been in corrosion control work for more than 20 years, principally in the formulation and application of phenolic type coatings.

J. E. PRIOR—Metallurgist engaged in corrosion research with Socony Mobil Oil Company, Inc. at Research and Development Laboratory, Paulsboro, New Jersey. Before coming to Socony Mobil in 1946, he was a physical metallurgist with an aircraft company and a copper refinery. He received a BS in metallurgical engineering from Lehigh University in 1934.

WELLON T. ROSE—Engineer for Commonwealth Edison Co., Chicago, he has a BS in electrical engineering from Clemson College. He has been engaged actively in corrosion control since 1942 except for naval service of 30 months. He is a member of NACE.

EDWARD SCHASCHL—Pure Oil Company, Research and Development Laboratories, Crystal Lake, Illinois. He received a BS in chemical engineering from Illinois Institute of Technology in 1945 and joined Pure Oil Company in 1948 as a process engineer. He is presently doing research in metallurgy and corrosion.

HOWARD D. SEGOOL—Technical manager of Polyken Sales Division, The Kendall Co., Chicago, he completed undergraduate work at Brown University as a chemistry major and took advanced degrees in organic chemistry at Yale University. He spent 10 years in the chemical industry in research, product and process development before joining Kendall Co. in 1948.

HOBART SHIELDS—Senior chemist with the Research Department, Standard Oil Co. (Indiana), Sugar Creek, Mo. refinery, he was graduated from University of Kansas in 1923. A member of NACE and ACS, before joining Standard he worked for Missouri, Kansas and Texas Railroad as a water treating plant supervisor.

J. W. SJOBERG—Chemical engineer engaged ing corrosion research with Socony Mobil Oil Company, Inc. at its Research and Development Laboratory, Paulsboro, New Jersey. He received a BS from Missouri School of Mines and Metallurgy in 1944. After serving two years in the Navy, he received an MS in chemical engineering from the same school. He started with Socony Mobil in 1948 at its Augusta, Kanas, refinery, and transferred to the laboratories in 1954. He is a member of ACS and Sigma Xi.

RONALD V. SKOLD—Research Associate, Illinois State Water Survey, Urbana, he has been engaged in research on corrosion of water distribution mains since 1954. He has a BS, chemical engineering, University of Illinois (1949). Between 1949 and 1952 he did research in the field of adsorption at Mellon Institute and from 1952 to 1954 was with Dearborn Chemical Co. and Aquamatic, Inc. specializing in ion exchange applications.

F. A. SMITH—Since 1947 he has worked at Argonne National Laboratory, principally on projects associated with liquid metal cooled EBR-I reactors and EBR-II reactor design. He has a BS in physics from Roanoke College, Salem, Va. (1943) and worked for Eastman Kodak Company during World War II as a physicist on the Y-12 electromagnetic separation plant, Oak Ridge, Tenn.

L. V. SORG—Chief chemist in the Research Department, Standard Oil Co. (Indiana), Sugar Creek, Mo. refinery. He has BS and MS degrees from University of Kansas and joined Standard in 1930. Holder of numerous patents, he is author of papers in the field of analytical chemistry. He is a member of Sigma Xi, RESA, ACS and AIChE.

MILTON STERN—On the staff of Electro Metallurgical Company, he has a BS from Northeastern University (1949) and SM and PhD degrees from Massachusetts Institute of Technology in 1950 and 1952. He was an Aluminum Company of America fellow and a Wierton fellow during graduate work done under direction of H. H. Uhlig. He remained a research associate after graduation until joining his present employer.

R. L. STUTZ—He has worked principally in the field of water treating, with special emphasis on corrosion control since joining the Research Department of Standard Oil Co. (Indiana) in 1956. He has a BA and MS from University of Kansas. He is a member of AAAS, ACS and the Royal Chemical Society of London.

L. P. SUDRABIN—Consultant to Electro Rust-Proofing Corp., Belleville, N. J. Mr. Sudrabin spent several months during 1956 and 1957 consulting on corrosion problems on petroleum production and refinery equipment and ships in Lebanon, Syria, Jordan, Saudi Arabia and the Caribbean. Before joining Electro Rust-Proofing he worked for Dorr Company, Dayton Power and Light Co. and Boston Edison Co. He is a member of numerous engineering organizations, including NACE in which he is active in technical committee work.

LEO J. WALDRON—A chemist in the metallurgy division of the U. S. Naval Research Laboratory, Washington, D. C. He has a BS and MS in chemical engineering from Michigan State University (1926 and 1928) and since then has been with the navy, National Bureau of Standards and Public Housing Adminstration doing chemical and metallurgical work. With NRL has worked nine years on cathodic protection of naval vessels.

KENNETH R. WALSTON—He has been engaged in corrosion studies for the Refinery Engi-

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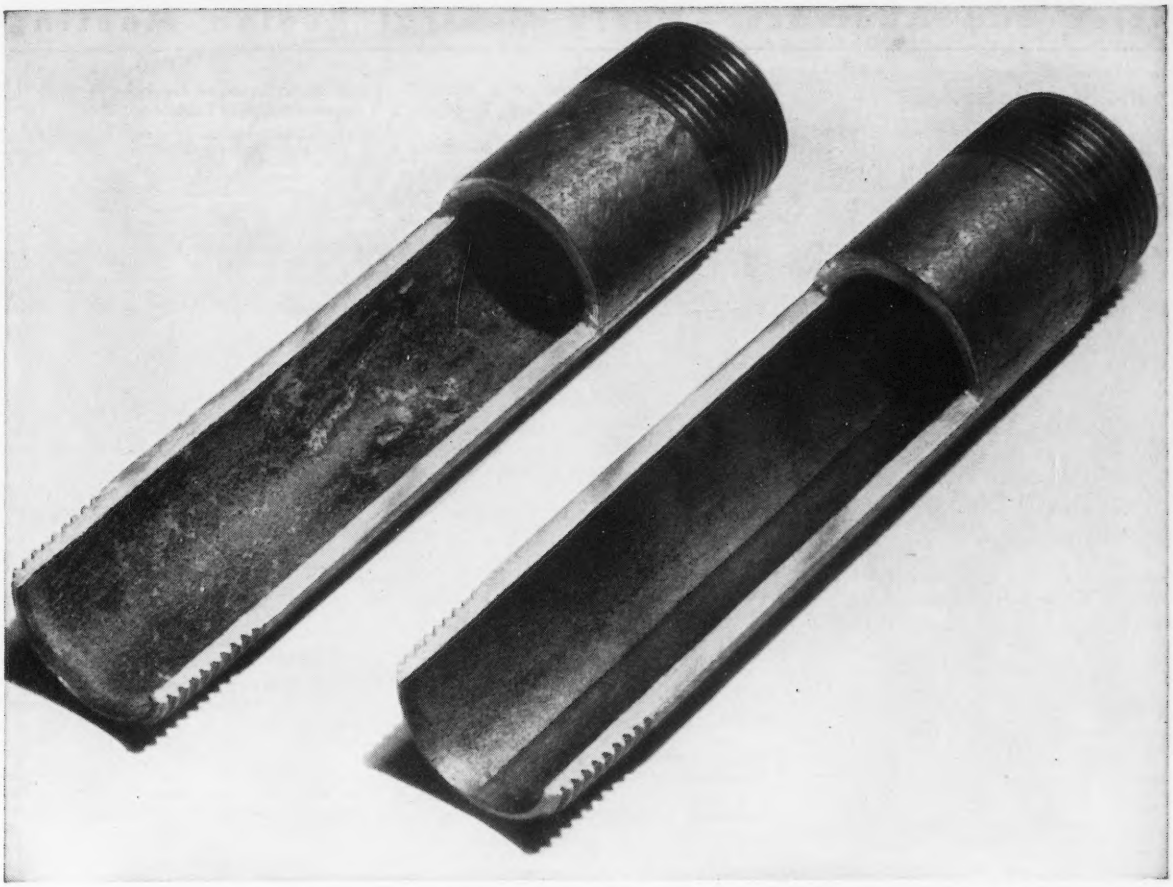

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Titles and Abstracts—North Central Region Meeting

Cathodic Protection

Experimental Studies of Cathodic Protection of Naval Vessels by L. J. Waldron, M. H. Peterson, and M. C. Bloom, Consultant Staff Corrosion, Metallurgy Division, U. S. Naval Research Laboratory, Washington 25, D. C.

This paper outlines a continuing experimental program of work on cathodic protection of naval vessels started in 1951. It discusses the many technical problems associated with the application of cathodic protection to active and inactive vessels, steps taken to solve these problems and future work necessary for further refinements in procedures and control.

Experimental details and results obtained after several years of operation are given for cathodic protection installations on 25 laid-up (Reserve Fleet) vessels at five locations. Results of this work have been used by the Bureau of Ships as a basis for development of standard specifications for installation of cathodic protection systems on all reserve fleet vessels.

Several cathodic protection systems involving both galvanic anodes and impressed current installed on active naval vessels are described. The various difficulties encountered in this work and the essential requirements for successful operation are outlined.

Measurement of Cathodic Protection, L. P. Sudrabin, Electro Rust-Proofing Corp.

Special techniques sometimes must be developed and adapted to measure effectiveness of cathodic protection on pipelines, process equipment, ship internals and hulls and marine structures.

Some considerations that must be made in the criteria used to significantly measure the corrosion control obtained include: a. Structure to reference electrode potential. b. Chemical examination of products on cathode. c. Comparison of structure thickness using nondestructive methods. d. Visual and physical examination of test panels. e. Pickup of corrosion product in the electrolyte.

Measurement of cathodic protection effects on the passive films of stainless steels and aluminum are discussed.

Evaluation of Protective Coatings Under High Potentials, Donald F. Koenecke and Robert I. McDougall, Esso Research and Engineering Co., Linden, N. J.

Since it is becoming a customary practice to use a combination of cathodic protection and protective coatings for corrosion mitigation, an accelerated test to evaluate protective coatings under these conditions is desirable. After a review of reported methods, an electrolytic test cell technique was selected as a versatile apparatus for laboratory screening and long term evaluations.

The cell set up consists of a holiday-free thin (2-5 mils) coated steel panel sealed on one end of a glass cylinder and a graphite electrode plate on the other. Cell electrolytes used are either N/2 NaCl, 10% H_2SO_4, or 10% NaOH. Using a 6-volt or higher DC current impressed across the cell with the coated panel as the cathode, film degradation is followed, observing current flow, capacities index and panel condition. Comparative data have been obtained on about 200 test panels prepared from 15 basic type coatings, some of which show greater than 100-fold differences in behavior. Effects of variables on the test will be discusssed.

Theory of Cathodic Protection, W. J. Kretschmer, Columbia Gas System Corp., Columbus, Ohio.

A presentation of the theory of, or mechanism of cathodic protection previously presented by R. B. Mears and others is made including in a practical manner the effect of cathodic or impressed current on corrosion current, anode potentials and current and cathode potential and current.

Galvanic anode polarization and its effect on cathode protection requirements will be considered generally.

Theory presented will be correlated with practical application of cathodic protection to pipelines particularly those in the Appalachian area.

Chemical Industry

Corrosion in the Corn Wet Milling Industry, Richard Benes, Fred J. Holsinger and Russell E. Pierson, Corn Products Refining Co., Technical Division, Argo, Ill.

Use of dilute sulfurous acid for steeping corn prior to milling results in numerous corrosion problems. Corn syrup, dextrose, corn oil and special starch production using such raw materials as sulfuric acid, hydrochloric acid, mineral carbon and sodium hypochlorite make it extremely important that the proper construction material is selected. The occurrence of stress cracking of evaporator tubes and selection of materials for various valves are discussed briefly. The use of stainless steels, sheet linings and specialized organic finishes have proved effective in combating the more serious corrosion and abrasion problems.

Corrosion Problems in the Manufacture of Phosphoric Acid From Elemental Phosphorus, J. C. Barber, Office of Chemical Engineering, Tennessee Valley Authority, Sheffield, Ala.

Principal materials of construction and equipment used for the production of phosphoric acid (56-58 percent P_2O_5) from elemental phosphorus are described. The process involves the combustion of phosphorus to form P_2O_5; the P_2O_5 is then reacted with water to form phosphoric acid. General applications of various metals, ceramics and plastics for use in constructing the processing equipment are discussed. High temperatures, which are characteristic of the process for the production of phosphoric acid from elemental phosphorus, cause severe corrosive conditions in parts of the system and impurities in the phosphorus burned and erosion intensify them. Tests and plant experience have indicated what steps might be taken to cope with the corrosion problems.

A highly concentrated phosphoric acid (76 percent P_2O_5) can also be made in the phosphoric acid production facilities. The corrosion problem incurred by the production of this acid is given.

Accelerated Condensation Corrosion Test for Evaluating Rust Preventives, E. A. Dieman and J. W. Gaynor, Research Dept., Standard Oil Company (Indiana).

Investigations have shown that the poor reproducibility of data in conventional humidity cabinet tests is due largely to: 1. Variable condensation of water on test panels and 2. Nonuniform surface condition of the test specimens. A new test, the Accelerated Condensation Corrosion Test (ACCT), has been developed which resolves these disabilities. Uniform and accelerated condensation of water on the test panels is obtained by substituting specimen "boxes" for the normal flat specimens and circulating cooling water through the boxes. A more reproducible panel surface is obtained by immersing the sand-blasted panels in water for a short period prior to the conventional alcohol rinse and rust preventive application. The beneficial effects of the water dip are attributed to formation of a more uniform iron oxide and hydrate film. Operating at a temperature differential of 30 F between the cabinet atmosphere and the cooled test panels, the standard deviation of ACCT data is about 13% as against about 65% in JAN-H-792 tests at Whiting. The time for initial rusting for products thus far tested has ranged from 1/25 to 1/80 of that obtained in conventional cabinet tests. Accelerated conditions make the ACCT particularly attractive for research studies as well as evaluation of quality of plant products.

Liquid Metal Corrosion Experience, F. A. Smith, Argonne National Laboratory.

By using liquid metals as a coolant it is possible to extract considerable heat/vol from an industrial process. This paper presents corrosion experience resulting from the use of NaK and Na as a nuclear reactor coolant. Temperatures and environment that both minimize and maximize liquid metal corrosion is considered. Experience gained in operating a Na loop of an engineering size at 1500 F is illustrated. Using high temperature as an ultimate criterion in the application of liquid metals as coolants, it is concluded that the understanding of the nature of corrosion in this field is not complete.

Coatings

Underground Corrosion and Its Mitigation by the Use of Coal Tar Coatings, F. J. Kiernan, Barrett Division, Allied Chemical & Dye Corp.

The paper covers the fundamental principles of underground corrosion, general methods of mitigation and history of coal tar protective coatings for underground use. Development and characteristics of hot and cold coal tar coatings are enumerated. Five general types of hot-applied coal tar coatings, as developed and used especially on pipelines, is treated at length. The paper also covers design requirements of the coal tar enamels based on environmental and service dictates.

Protective Coatings in Use With Cathodic Protection, F. Ploederl, Wisconsin Protective Coatings, Inc.

Classifying Protective Coatings and How to Evaluate the Base Resin Formulation for Maintenance Purposes, L. Gleekman, Wyandotte Chemical Co.

Manufacture, properties and methods of using generic types of resins in maintenance painting are discussed. Formulation evaluations will be considered in terms of analysis of the resin, paint formulation, laboratory tests (including salt spray and exposure tests) and finally full scale plant tests. Emphasis is placed on correlation of coating performance with environmental conditions. Several current paint testing programs will be explained and a practical program for evaluation of coating performance on equipment in service will be outlined.

Chemically Preparing Metal Surfaces for Coatings, John N. Geyer, American Chemical Paint Co., Ambler, Pa.

Commercial Spray Equipment, Including Hot Spray, Heavy Duty Materials, Low Pressure and High Pressure Spraying and Equipment Maintenance, W. R. Brooks, Binks Mfg. Co.

Educational Lectures

Electrochemistry of Corrosion—Some New Ideas, Milton Stern, Electro Metallurgical Co.

Advances made in the theory of electrode kinetics can be applied to corroding systems and supply a clearer understanding of the many factors which affect the corrosion rate of a metal. A modified polarization diagram is described and is used to illustrate how a number of environmental changes affect corrosion rate. Galvanic couples also are discussed in terms of activation and concentration polarization.

Some Unusual Effects of Hydrogen in Corrosion Reactions, J. E. Draley, Argonne National Laboratories.

It is postulated that hydrogen produced when certain metals are corroded by water acts to damage the protective layer covering the metal surface. It is considered that some hydrogen ions diffuse through the layer, forming hydrogen atoms. The consequence of this action is

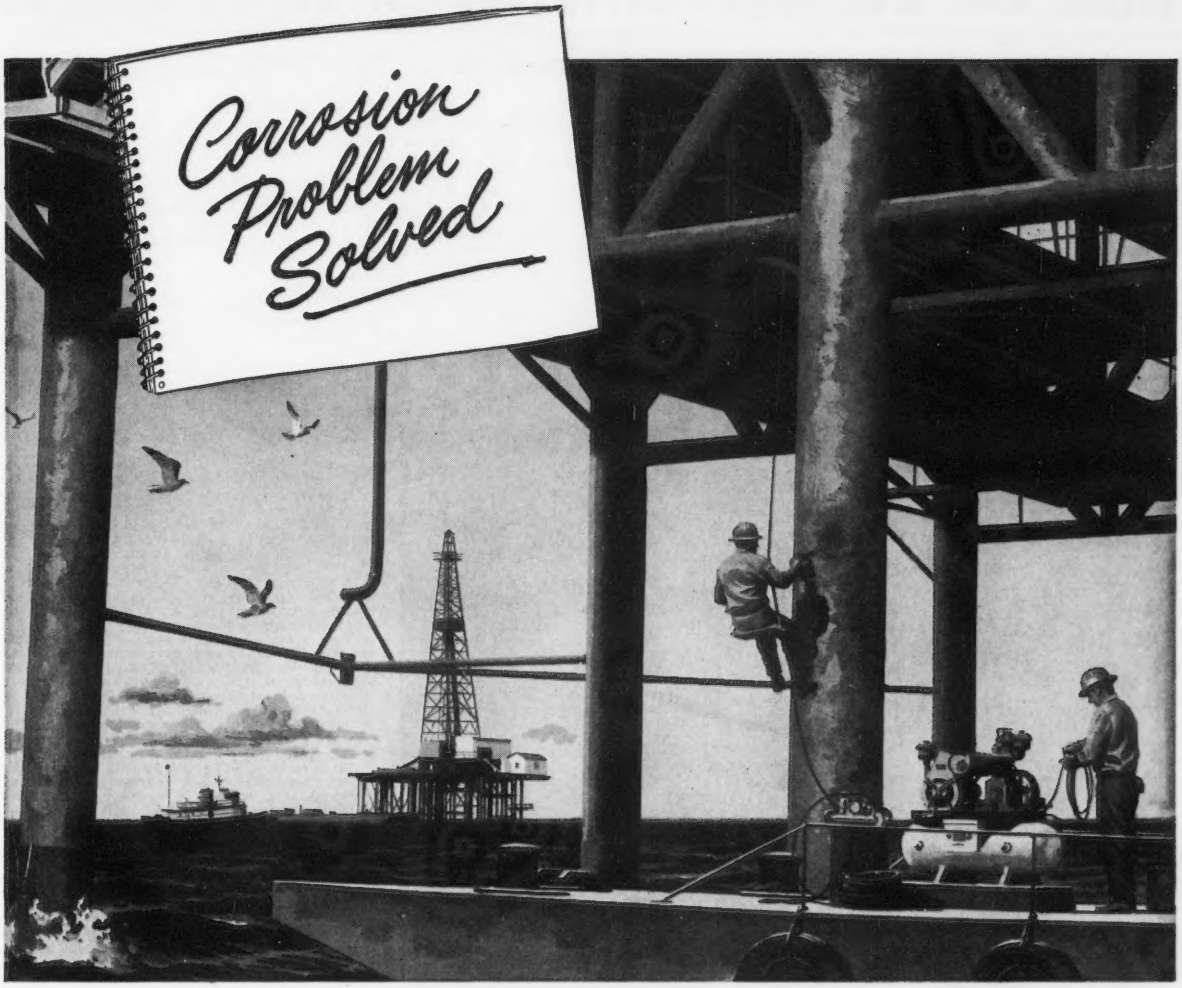
(Continued on Page 104)

BIOGRAPHIES

(Continued From Page 100)

neering Division, Standard Oil Company of Indiana since 1953. He also has coordinated chemical cleaning activities at the Whiting, Ind. refinery of his company. He has a BS in chemical engineering from University of Illinois, 1950 and joined his present employer after service in the U. S. Army's Chemical Corps.

FRANKLIN M. WATKINS—Director of the Corrosion Division, Sinclair Research Laboratories, Inc., Harvey, Ill. He has been concerned with the development of additives for petroleum products, an outgrowth of his interest in transformer oils developed while with General Electric. He spent a year investigating tanker corrosion problems for American Petroleum Institute's Transportation by Water Committee. He is active in NACE.



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Titles and Abstracts—North Central Region Meeting

(Continued From Page 102)

to decrease corrosion resistance. Experiments with aluminum, zirconium and uranium are described.

Electrochemical Mapping of Corroding Surfaces, H. T. Francis, Armour Research Foundation.

Existence of local anodic and cathodic zones on a corroding surface has long been accepted as an essential feature of the electrochemical theory of corrosion. Only rarely, however, has it been possible to observe such zones directly. A technique is described in which a corroding surface is scanned by a reference electrode and the electrical information presented on an oscilloscope as an "electrochemical map" of the surface. In one form of the apparatus, a black and white "television picture" is produced in which anodic zones are bright and cathodic zones dark, respectively.

Pipe Line Symposium

Internal Corrosion Inhibitor Testing on a Products Pipe Line, Robert H. Meyer, Sinclair Pipe Line Co., Independence, Kans.

Observations and data obtained during testing of four different oil soluble internal corrosion inhibitors in a 151-mile section of a products pipe line are discussed in detail. Seven evaluation procedures used were: 1. Line test coupons; 2. Product filtration; 3. Specially prepared test segments of pipe; 4. Modified turbine type rust tests; 5. "C" factor or roughness factor; 6. Scraper trap deposits; and 7. Frequency of strainer plugging. The latter two methods are relative methods because they are especially subject to changes in operating conditions. The test coupon method of evaluation is the most desirable because of its simplicity and reliability.

A very slight depletion of inhibitor in the product after traversing the 151 miles was generally indicated even though a high degree of over-all protection was maintained. One of the inhibitors tested produced a corrosion product of a more bulky and amorphous structure which caused strainers to plug more rapidly even though the over-all rate of corrosion was low.

Analyses by X-ray diffraction methods of the material deposited on the filters was found to be a method of determining the relative amount of newly formed corrosion products in the pipe line during the tests of the various inhibitors.

Asphalt Coatings for Pipeline Protection, David C. Palm, Natural Gas Pipeline Co. of America.

A series of tests were made on an asphalt coating for pipelines to appraise three basic properties: 1. Its effectiveness as a barrier against the passage of moisture and electrical currents. 2. Does it permit a quality standard of field application? and 3. Evaluation of its lasting qualities. As a result of these tests the coating gave promise of being satisfactory and was applied to 30 miles of 36-inch pipeline.

The fiber glass reinforced asphalt coated line was laid in areas of clay, loam and limestone rock soil in the northern part of Illinois. Application procedure was typical of that used in the general category of bitumen coatings and there were no serious application difficulties.

Experience on this job indicates there is some saving in the cost of this coating in place when compared to the cost of other bitumen coated projects built in recent years.

Two years after installation, exhaustive tests were made on coating performance. The bond between the pipe wall and coating was good. Efficiency of the cathodic protection system compared favorably with other installations on bitumen coatings which are rated as good by affiliated companies. Observation indicates that no detrimental absorption of moisture has taken place.

It is early to draw conclusions regarding the lasting qualities of any coating job after only two years of service. Some very slight deformation of the coating from soil stress could raise the question whether it is as tough as other coatings which have stood up well for many years under very unfavorable soil conditions.

Corrosion Protection for Pipe-Type Transmission Lines, Wellon T. Rose, Commonwealth Edison Co., Chicago.

In the Chicago area about 30 miles of pipe type high voltage transmission lines has been placed in service. These pipes are protected with either an asphalt mastic coating or a reinforced coal tar enamel coating. Both are factory applied.

Cathodic protection is applied to these lines to police the pin holes in the coating. Before cathodic protection could be economically applied, it was necessary to insulate the pipes from the station ground busses. It was also necessary to maintain a low resistance path between the pipes and the ground busses for fault current in the event of a service failure. This was accomplished by using oxide film cutout disc in the ground connections. Both magnesium anodes and a rectifier are used to provide cathodic protection.

Tape Coatings in Pipeline Construction, Howard D. Segool, Polyken Products, The Kendall Co., Chicago.

Tape Coatings based on pre-fabricated synthetic plastic films bearing a bonding adhesive have assumed an important place in the pipeline protection field. Deriving from the accepted use of adhesive plastic electrical insulation tapes, tape coatings had their start as accessories in the protection of welded joints, small pipe and fittings. Development of application machinery and a good performance record advanced their applicability to use as total pipe coatings. These reproducibly uniform coatings, cold applied with simple equipment, minimal labor and great construction efficiency now compete with older melt-type coatings on a quality-at-no-premium basis.

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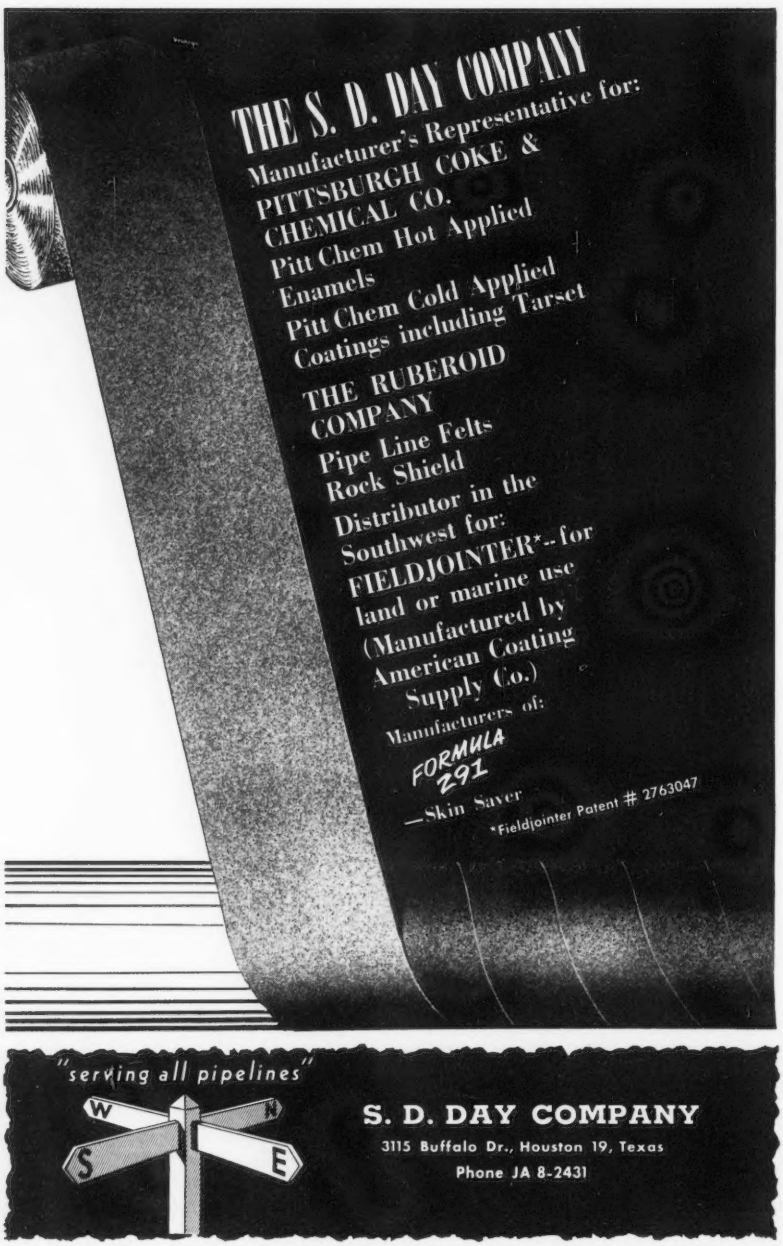

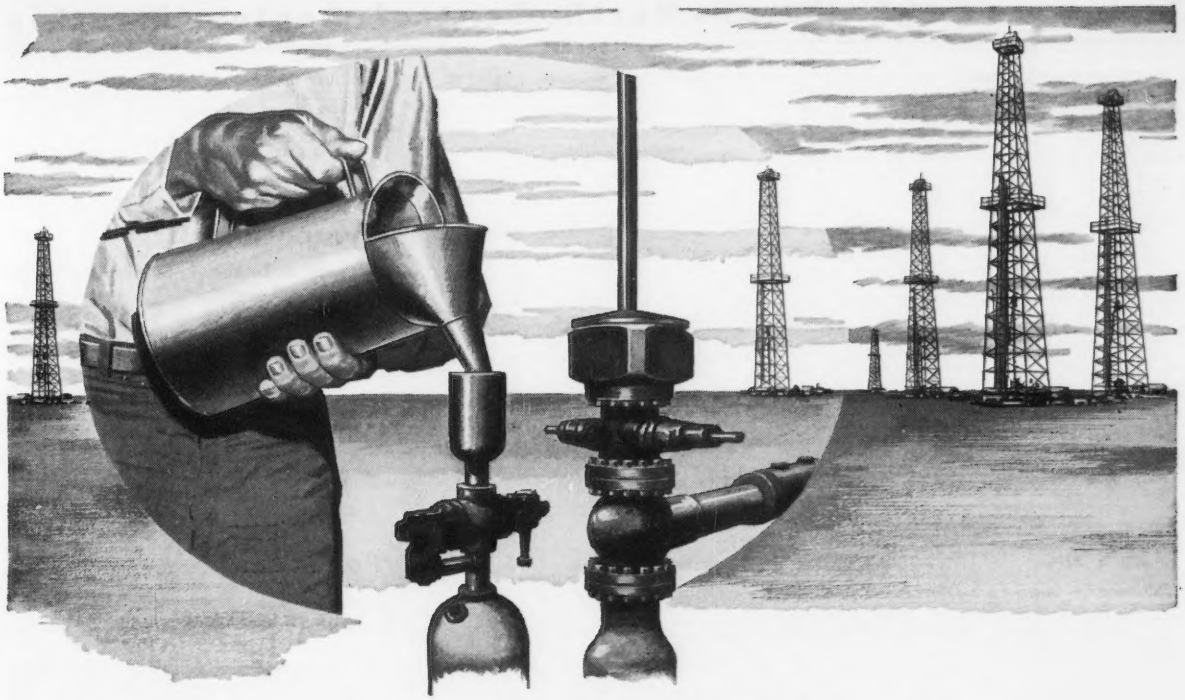

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Write for a copy of the new booklet, "Cronox Corrosion Inhibitors."

Titles and Abstracts—North Central Region Meeting

(Continued From Page 104)

Refinery Symposium

High Temperature H_2S Corrosion of Stainless Steels, E. B. Backensto, R. D. Drew, J. E. Prior and J. W. Sjoberg, Socony Mobil Oil Co., Inc., Paulsboro, N. J.

In recent years the petroleum industry has been plagued with a number of high temperature H_2S corrosion problems, primarily in connection with catalytic reforming. To combat this problem from a standpoint of materials selection, only the austenitic steels offer satisfactory resistance to this type of attack under a wide range of conditions.

This paper reports the results of laboratory investigations chiefly on stainless steels, covering the following main topics: 1. The possible application of the new manganese-modified stainless steels, 2. The effect of heat treatment on corrosion rate, 3. A comparison of corrosion rates of wrought, weld and cast stainless steel, and 4. Some tests on stress corrosion cracking.

Corrosion of Refinery Equipment During Acid Cleaning, K. R. Walston and A. Dravnieks, Standard Oil Company (Indiana).

Hydrochloric acid is the most widely used solvent for chemical cleaning of refinery equipment because it is economical, effective and relatively easy to handle. Its strong acid properties when combined with the surface activating tendencies of the chloride ion can result in rapid corrosion unless proper procedures are followed. It must be inhibited to prevent corrosion of the metal but the inhibitor must not excessively retard the dissolution of scales. The practical maximum operating temperature for acceptable limits of corrosion appears to be 170 F although this will vary somewhat depending upon the inhibitor used.

Laboratory test work on several cleaning acids indicate that the corrosion rate varies exponentially with increasing temperature and very nearly exponentially with concentration. When hydrogen sulfide is present in the cleaning solution, the corrosion rate is increased two to three-fold. Ferrous and chromic ions in addition to the widely recognized ferric ion greatly accelerate the corrosion rate of mild steel. Free machining steels were found to corrode 30-100 times faster than low carbon steels. Severe corrosion also has been experienced with 12% Cr. trim in steel gate valves, particularly in the crevice presented by the disc and the seats of the valve. Crevice corrosion also has been encountered during the pickling of furnace tubes for mill scale removal. Some additives appear to reduce these crevice effects unless hydrogen sulfide is present. Rapid agitation also appears to lessen this crevice attack. In view of the increased consumption of hydrochloric acid for refinery cleaning especially where higher concentrations and temperatures are involved increased diligence is warranted to insure that hydrochloric acid used for cleaning equipment is handled with the respect it deserves.

Corrosion in Phenol Extraction Units, G. A. Marsh and B. Schaschl, Pure Oil Co.

Brittle Characteristics of Vessel Steels After 28 Years in Thermal Cracking Service, F. B. Hamel, Standard Oil Co. of Ohio, Cleveland.

A low temperature brittle type crack eight feet long which occurred in December, 1954, led Sohio into this investigation. Twenty steel plate samples were taken from eight vessels of a thermal cracking unit. The tests on these samples included impact, chemical, physical and micro-structure tests. All samples were brittle at ordinary winter temperatures.

Computer correlation work was done in an attempt to find out if operating temperature or time-in-service had lowered the impact resistance. There is some indication that operating temperatures decrease the impact resistance—time-in-service seems to have no effect. Correlation with published data was not good. By use of some additional data, an explained variance of 80% was obtained.

While these results are significant in themselves, additional data are needed to obtain a complete picture of the brittle characteristics of older equipment. This condition also exists in current vessel steels. Sohio's work confirms the fact that the chemistry of a vessel steel has a major influence on its impact quality. Perhaps pressure vessel and tank steels could be produced to specifications similar to the American Bureau of Shipping specifications which obtain better impact qualities without impact testing.

Transportation Symposium

Corrosion in Tankers, F. M. Watkins, Sinclair Petroleum Laboratories.

Laboratory Testing of Railroad Diesel Cooling System Corrosion Inhibitors, J. I. Bregman and D. B. Bores.

Some Aircraft Corrosion Problems, H. L. Stevens, Wright Air Development Center.

Conductivity Testing of Zinc Dust Pigmented Coatings. Lt. D. S. Nantz, Wright Air Development Center.

Public Utilities

Cathodic Protection of Electrical Generating Stations, A. W. Peabody, Ebasco Services, Inc.

Use of a Magnetic Amplifier for Variable Rectifier Output, Harry E. Kroon, Illinois Bell Telephone Co.

This paper covers a field application of a magnetic amplifier used to vary the rectifier output simultaneously with the erratic positive cable-to-earth potentials.

Use of Plastic Tubing for Replacements in Low Pressure Gas Distribution in Chicago, T. R. Bogumill and Hugh R. Murphy, Peoples Gas Light and Coke Co., Chicago.

This paper deals with the experience of the Peoples Gas Light and Coke Company with plastic tubing for insertion renewals of defective steel service piping. It comments on the experiments and research conducted to determine which of the many plastics available would be most suitable. Factors which influenced the final selection will be discussed. Standard quality control checks of new shipments of plastic will be explained and a brief summation given of recommended Plastic Pipe Standards, as proposed by the Subcommittee on Plastic Pipe Standards of the American Gas Association.

Soil Box Demonstration, M. C. Miller, Ebasco Services, Inc.

Water Treating Symposium

Aluminum Cooling Towers and Their Treatment by A. J. Haygood and J. Dean Minford, Aluminum Company of America, New Kensington, Pa.

Many industrial processes require tremendous quantities of water to remove heat from operating equipment. Economic considerations and municipal restrictions often make it necessary to recirculate the water through a cooling tower. Aluminum, because of its light weight, good resistance to corrosion and potential low fabricating and maintenance costs is being considered as a material of construction for equipment handling these recirculated waters. Some natural waters offer unique corrosion problems; and, also, because of corrosion, associated with multimetallic systems, water treatment is becoming standard practice. In general, then, each system should be considered separately as to design, operation and water treatment.

Aluminum cooling towers will be considered with respect to proper alloy selection, fabricating procedures and surface finishing. Water treatments utilizing borates, citrates, chromates, nitrates, nitrites, mercaptobenzothiazole, phos-

(Continued on Page 108)

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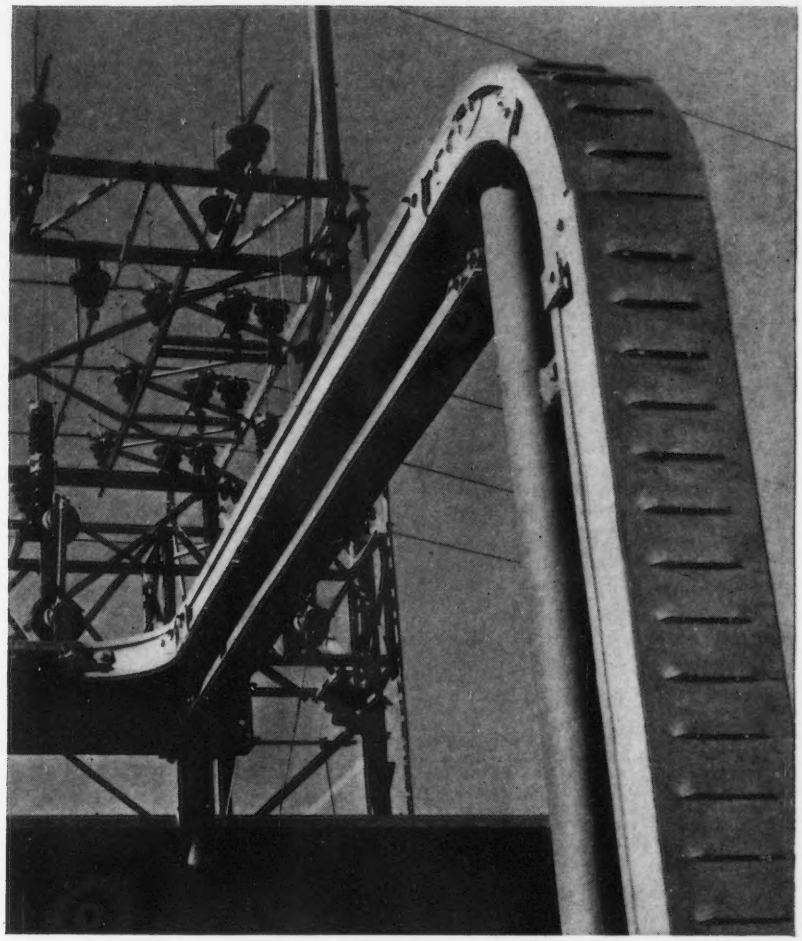

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Titles and Abstracts

(Continued From Page 106)

phates and silicates and combinations of these chemicals have been used successfully in multimetallic systems. Examples of these installations will be given and the results of field experience cited.

Selecting Corrosion and Scale Inhibitors for Cooling Water, H. Shields, L. V. Sorg and R. L. Stutz, Standard Oil Co. (Indiana).

Corrosion inhibitors for cooling water are subjected to four phases of testing: Bench-scale, pilot plant, small size commercial cooling tower and full plant-scale.

In bench-scale tests, mild steel coupons are immersed in aerated water at varying concentrations of inhibitor. Pilot plant testing is unique in that glass-cased heat exchanger units are employed. Metal penetration data from the pilot plant can be correlated with those obtained on a plant-scale; thus plant-scale performance can be predicted from tests conducted at the pilot plant level.

Promising inhibitors usually are tested in the small size commercial cooling tower using test units identical with those in the pilot plant. Inhibitors which fulfill the requirements of the above three tests then are considered for plant-scale application.

Over nine years, approximately 50 inhibitors have been investigated of which only three were given full-scale trial. Of these, only the chromate-type has proved satisfactory in all phases of testing. Organic chromates have been found to be particularly effective for the Missouri River water as used at the Sugar Creek refinery. Phosphates, while exhibiting fair corrosion protection, had the disability of depositing scale, even at low pH values.

Results of a Study on How Mineral Content of Water Affects Corrosion, T. E. Larsen and Ronald V. Skold, Illinois State Water Survey.

The influence of combinations of various normal mineral components in domestic waters on the rate of corrosion of cast iron and on the development of tuberculation is being studied. This report deals with air saturated waters at room temperature with velocities of 0.1-1.0 fps and with pH controlled by carbon dioxide. The ions considered are chloride, sulfate, bicarbonate and calcium at pH 6.8-8.3.

Young Author Maximum Age Reduced to 30 Years

The maximum age of authors competing for the Young Author Award, beginning with articles published in the January, 1958 issue of CORROSION has been set at 30 years by the NACE board of directors. The maximum age previously had been 36.

The board acted to reduce the age because of the large number of authors qualifying under the previous limit. For example, already during 1957 there are 52 authors under consideration. It also was a consensus that reducing the age might encourage more contributions to CORROSION from students and graduate students in universities.

The award, established in 1952 with funds supplied by an anonymous donor, consists of a gratuity of $50 to the author whose paper published in CORROSION during the calendar year is judged best among those competing. If co-authors are involved the gratuity is divided among them. Decision on the best paper is made by a committee named annually by the NACE president. Since the award was first made, the Education Committee has been named to do the judging.

Authors interested in the award may get on request a copy of the NACE Guide for Preparation and Presentation of Papers, which is helpful in describing the needs of the association for technical papers and the manner of their preparation.

GENERAL NEWS

Los Angeles Air Pollution Controls Cost $298 Millions

The value of basic equipment installed since 1948 to control air pollution in Los Angeles County, California has risen to $298,773,400 by the addition of $18,807,600 during April of this year.

Citations for violations of air pollution control laws increased 11 percent for the first three months of 1957 compared to the same period last year; fines were up more than 300 percent, one repeater truck offender being fined $500, and two persons previously repeatedly convicted of open burning were sentenced to 30 and 60 days in county jail with the balance of 180-day suspended on good behavior.

New laws enacted by the California legislature give the Air Pollution Control District of Los Angeles authority to cite smoking vehicles under the State Vehicle Code as well as under Health and Safety Laws.

The district also has new power over sale of auto exhaust devices and may require permits for the sale of equipment manufactured outside the county. Weed burning by public agencies also is restricted and government jurisdictions brought under air pollution control laws and regulations.

Pipeline Coatings Topic At Tulsa ASME Session

The three-day Twelfth Annual Conference of the Petroleum Division, American Society of Mechanical Engineers will be held at Hotel Mayo, Tulsa beginning September 23. More than 40 papers will be given and a discussion on the ASME pressure vessel code will be held.

Scheduled in the session on pipe line coatings are:

Application of Plastic Tape to 122 miles of 22-inch Diameter Natural Gas Pipe Line, Norris E. Miley, American-Louisiana Pipe Line Co.

Development and Application of High Density Rubberized Asphalt Mastic Coating, L. N. Brown, Southern Natural Gas Co.

Application of Epoxy Coatings to Interior and Exterior of Large Diameter Pipe, Raymond Crowe, Transcontinental Gas Pipe Line Corp.

Zirconium and Lithium Standards to Be Published

ASTM standards on zirconium and lithium will be published during 1958 by Committee B-2 on Non-Ferrous Metals and Alloys. Groups are being organized to consider standards for beryllium, columbium, tantalum, thorium, uranium and hafnium and molybdenum and tungsten metals and alloys.

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Abstracts in CORROSION come from 28 agencies.

BOOK NEWS

Electrochemical Behavior of Boron. Diagrams of Equilibrium Tension-pH of the System B-H_2O at 25 C. By E. Deltombe, N. de Zoubov and M. Pourbaix. **Electrochemical Behavior of Bismuth.** Diagrams of Equilibrium Tension-pH of System Bi-H_2O at 25 C. By J. Van Muylder and M. Pourbaix. (In French.) 22 pages, 8½ x 11¾ inches, paper. March, 1957. Cebelcor, 21 Rue des Drapiers, Brussels. Per copy, francs 170.

Annual Report 1956 of Dechema. (In German.) 8½ x 11½ inches, paper. July, 1957. Dechema, Frankfurt-am-Main 7, Germany. Free.

Filler Metal Comparison Charts. AWS A5.0-57. 23 pages. 11 x 8½ inches, paper, July, 1957. American Welding Society, 33 West 39th St., New York 18, N. Y. Per copy, $2.

A comparative list of the filler metal electrodes of 61 manufacturers are listed so a comparison may be made among the brands in 12 AWS-ASTM classifications. An alphabetical list of brand names is included.

The Industrial Chemistry, Properties and Application of Silicones by Charles E. Reed. 47 pages, 6 x 9 inches, paper. 1956. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. Per copy, $1.50.

The Edgar Burburg Lecture, 1956.

Guide to Atomic Energy Literature for the Civilian Application Program. TID 4575. 74 pages, 6 x 9 inches, paper. April, 1957. Technical Information Service Extension, AEC, P. O. Box 1001, Oak Ridge, Tenn. Free.

A description of available information materials, methods of locating information and how to obtain technical materials information and services. Volumes of the National Nuclear Energy Series and other publications are listed in indices.

Reinforced Plastics Meeting

Reinforced Plastics Division, Society of the Plastics Industry, Inc. will hold its 13th Annual Technical and Management Conference February 4-6 at Edgewater Beach Hotel, Chicago. The program includes sessions on strength-time behavior and other factors.

15-Week Paint Course Is Scheduled at New York U.

Basic principles applicable to the manufacture and application of paint will be subject matter of a 15-week evening course at New York University's Division of General Education this fall. The class will meet Wednesdays beginning September 25.

Meetings on Nuclear Fission Scheduled for October and March

Two meetings to be devoted to matters involved with nuclear fission have been scheduled for October in New York City and in Chicago in March, 1958.

The Second Winter Meeting of the American Nuclear Society will be held at the Henry Hudson Hotel, New York City October 28-31. It will be concurrent with the Fourth Annual Meeting of the Atomic Industrial Forum and with the 3rd Trade Fair of the Atomic Industry at the New York Coliseum.

The 1958 Nuclear Congress to be held in Chicago includes a number of subsidiary meetings, including the 4th Nuclear Engineering and Science Conference, 4th International Atomic Exposition, 6th Atomic Energy in Industry Conference, 6th Hot Laboratories and Equipment Conference and the American Power Conference. Some 30 trade and engineering groups are cooperating.

Gleekman to Give Detroit Graduate Corrosion Course

A graduate course in corrosion mechanism and mitigation will be presented during the fall semester at Wayne State University, Detroit, according to the Educational Committee of NACE's Detroit Section. L. W. Gleekman, manager of Materials Engineering Group, Wyandotte Chemicals Corp., will present the course.

All interested in corrosion may attend. Further information can be obtained either from Dr. Gleekman or from H. G. Donnell, Head, Chemical and Metallurgical Engineering, Wayne State University.

Plastic Roofing Sessions Set at Washington U.

Plastics for Roof Construction is the theme for a September 17-18 meeting of the Building Research Institute's Plastics Study Group at Washington University, St. Louis. A registration fee of $20 covers all sessions, two luncheons, bus fare and a copy of the report of the meeting.

Some of the subjects scheduled are: Vinyl Film as Vapor Barrier, Roof Structures Constructed of Plastic Material, Plastics and Elastomer Modified Asphalts, Neoprene as a Roof Coating, Thermosetting Plastics Skylights.

New ASME Secretary

Oscar B. Schier II has been designated secretary-elect of The American Society of Mechanical Engineers succeeding Clarence E. Davies, retired after 23 years.

Equipment Services NEW PRODUCTS Materials Literature

Atomic Energy

Nuclear Fuel Elements for atomic reactors will be manufactured in a new plant under construction by Sylvania-Corning Nuclear Corp. at Hicksville, N. Y.

Coatings

Amercoat Corp., 4809 Firestone Blvd., South Gate, Cal. has data sheets available on the following coatings: High Build Vinyl—Two coats over primer will exceed customary 5-mil thickness. Protects against numerous corrosives. Described in Bulletin 33HB. Decorative vinyl protects against chemical exposures outdoors. Described in Bulletin No. 35. Vinyl coating for masonry, described in Bulletin No. 88.

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Coatings for pipe, joints, fittings and couplings will be subjects of research in a new research and development department established by The Tapecoat Company, Evanston, Ill. Bernark Wolnak, president of Mid-West Laboratories heads the research staff. Expansion of research into general protective coatings is contemplated.

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Halofluorocarbon Dispersion Coatings manufactured by Minnesota Mining and Manufacturing Company's Jersey City Chemical Division, Chemical Products Group, P. O. Box 477, Jersey City, N. J. are described in two recent brochures available on request.

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Protection against fingerprints for polished surfaces is claimed for the film deposited by non-woven rayon silicone treated polishing cloths available from Edmund Scientific Co., Burlington, N. J.

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Zinc is used as a base plate in a new copper-nickel-chrome plating sequence developed by Bruno Leonelli, director of research for Wagner Bros., Inc., Detroit. The inventor claims this system produces a coating substantially superior to standard techniques presently used.

Construction

Canadian Badger Ltd., 25 Kind St. West, Toronto, Ontario has been organized as a company to design and construct oil refining, chemical, pharmaceutical, ore processing and paper manufacturing plants by Badger Manufacturing Co.

Fasteners

Stainless Steel Data is title of a booklet issued by Allen Manufacturing Co., Hartford 2, Conn. describing its Hex-socket screws.

Metals—Ferrous

High-Purity high alloy steels, zirconium, titanium and a wide range of ferrous and non-ferrous alloys can be melted in ingots up to 26 inches in diameter and weights up to 12,000 pounds in a new consumable electrode furnace put in operation at Watervliet, N. Y. by Allegheny Ludlum Steel Corp.

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Wrought Iron electrical conduit has been granted use of Underwriters' Laboratories, Inc. seal after a series of tests. A. M. Byers Co., Pittsburgh will make wrought iron rigid conduit with hot-dip galvanized or vinyl plastic coating in ¾ to 6-inch trade sizes.

•

Wrought Iron plates were used to fabricate the hull of 95-foot offshore crew boat for Begnaud Marine Service. Platzer Shipyard, Houston used the wrought iron plates at the insistence of the Begnaud Company, which operates out of Rockport, Texas. Copon coatings were used on upper works.

Metals—Non-Ferrous

Aluminum and aluminum coated steel pole line hardware is now being tested in industrial atmospheres to determine which has longer life. Pennsylvania Railroad and Kaiser Aluminum & Chemical Corp. are cooperating in the tests near Whiting, Ind. where standard galvanized hardware failed within three to five years. Hot dipped aluminum coated mild steel and low-alloy, high strength steel are being tested in comparison with standard galvanized steel and galvanized low-alloy high strength steel.

•

Aluminum pigs guaranteed to be not less than 99.5 percent pure are being produced by Aluminum Company of America. Alloy X5454, developed for structures operating at 150-300 F has high resistance to corrosion and stress corrosion. Welding characteristics are said to be excellent. Alloy 5456, rolled into plates, has the highest mechanical properties available in the nonheat-treatable alloys and besides this has good resistance to corrosion and stress corrosion. Welding characteristics are good. Creole Petroleum Corp. is installing 22 miles of oil-carrying aluminum pipe on the floor of Lake Maracaibo, Venezuela.

•

Apex Smelting Co. is full owner of National Metallurgical Corp. after purchase of the half-interest formerly held by American Smelting & Refining Co. The plant at Springfield, Oregon has been in experimental production of aluminum silicon alloys.

•

Boron, a basic ingredient in most high energy fuels is being produced in volume from borax deposits in Southern California, which has an estimated 95 percent of the world's known supply. American Potash and Chemical Company's Trona plant produces 25 percent of the annual boron tonnage and some 30 boron chemicals.

•

Titanium boat engine silencers may become common, according to Maxim Silencer Co., testing silencers on a twin-Diesel vessel. The water-cooled silencers are substantially lighter and more resistant to corrosion than those used presently.

•

Titanium Fact File, a 24-page publication of Mallory-Sharon Titanium Corp., Niles, Ohio, describes advantages of titanium over some competing materials.

•

Titanium-Base master alloy formulations used to promote ingot homogeneity in producing vanadium and aluminum-containing alloys, manganese-aluminum, tin-aluminum, tungsten aluminum, tantalum-columbium-aluminum and aluminum-molybdenum titanium alloys are available from Electro Metallurgical Company, Division of Union Carbide Corp., 260 Madison Ave., New York. The company also is a large supplier of sodium-reduction sponge titanium.

Inhibitors

Inhibitor CS for closed circulating water systems with or without antifreeze will protect steel, copper, brass aluminum and solder. It is available from Hagan Chemicals and Controls, Inc., 323 Fourth Ave., Pittsburgh, Pa.

(Continued on Page 112)

COKE BREEZE

Backfill for Anodes

Ideally suited for use with anodes. Has a high carbon content and comes in sizes of ⅛-inch x 0 to ⅜ x ⅞-inch. In bulk or sacks. Prices on other sizes on request.

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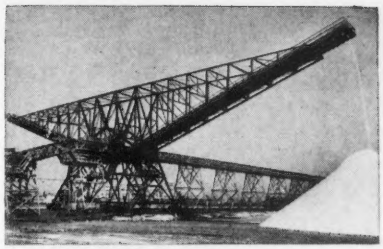
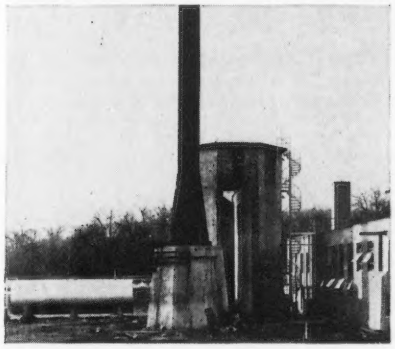


19 Years of Protection were given this giant gantry crane at a salt-producing plant by a coating system of Bitumastic Super Service Black. Exposed to the corrosive combination of salt plus atmospheric moisture, the coating system has required only minor touch-up during this service.



High Temperature Stack is protected against corrosion by a coating of Bitumastic® Hi-Heat Gray, a zinc-base coating designed for high-temperature protection. Intermittent temperatures up to 1000° F. and continuous 800° F. dry heat will not affect this coating material, and it is widely used for protection of boiler breeching, stacks and other hot metal surfaces.

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a report by Koppers Company, Inc.

Russell C. Stromquist
Corrosion Consultant

Recently, we were asked to recommend protective coatings in a plant on the West Coast. In analyzing their coating and enamel needs, we did our usual survey of the installation. After the survey, the customer's Chief Engineer remarked, "You fellows certainly go to a lot of trouble just to tell us what coating system we should use."

We do—because we know it is good for our customers and, therefore, good for us. Case history records prove clearly that careful study *before* making recommendations insures a better protective coating job at lower annual cost. In analyzing protective coating needs, our corrosion engineers follow a "pre-coating check list" based on experience on thousands of jobs.

* * *

The first step in the pre-application survey is a study of the environment. Specifically, you should always take into account the corrosive atmosphere: such factors as acid fumes, intense sunlight, salt air, etc. Physical environment—exposure to abrasion or impact—also plays an important part in the selection of an effective coating system.

The extent of temperature variation and expected minimum-maximum temperatures are important considerations, since the coating chosen must (a) resist low-temperature embrittlement, (b) resist high-temperature flow and (c) harden properly at service temperature.

The moisture environment too, is important in selecting protective coatings. Besides necessary water resistance, the coating should stand-up under pronounced wet-dry cycles.

A thorough cost analysis is a vital part of any pre-coating survey. Many factors are involved, varying in importance with the specific application. For example, total coating cost—made up of surface preparation and application costs—must be balanced against the cost of periodic maintenance. In some cases, the coated surfaces will not be accessible and coating-life will be a *direct* measure of the life of the material.

The cost of probable shut-down or loss of production time is perhaps one of the most important evaluations that must be made in intelligent selection of corrosion-preventing coating systems. Often, it outweighs the direct replacement costs of materials to be protected.

* * *

This pre-coating check list, detailing the protection required and under what circumstances, is a key factor in the exceptional performance of Bitumastic® coatings. Such thoroughness sometimes leads us to willingly recommend some material other than one of Koppers eleven basic Bitumastic coatings to solve a customer's problems.

* * *

In the next report, we will discuss why surface preparation is so vital to the life and economy of any protective coating. Meanwhile, we'd like to hear about your corrosion problems. Koppers Company, Inc., 1300 Koppers Bldg., Dept. 100J. Pittsburgh 19, Pa. District Offices: Boston, Chicago, Los Angeles, New York, Pittsburgh and Woodward, Ala.

KOPPERS

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COATINGS AND ENAMELS

NEW PRODUCTS

(Continued From Page 110)

Inspection

Radiographic Inspection of castings, welds, assemblies and repaired metal products is described in a folder available from Mitchell Radiation Products Corp., 128 East Washington St., Norristown, Pa.

Lubricants

Sexauer Faucet and Valve Stem Lubricant, a product of J. A. Sexauer Mfg. Co., 2503 Third Ave., New York 51, N. Y. retains normal texture under extremes of temperature, includes inhibitors which make it resistant to oxidation and is said to be non-staining and non-corrosive.

Non-Metallic Materials

Asphalt Roofing compounds perfected by The Panther Co., 840 N. Main St., Fort Worth, Texas are designed for application especially to concrete roofs or other types of built up or metal roofs. The petroleum asphalt material, according to manufacturers, retains adhesiveness and will last twice as long as other similar materials.

Unplasticized Polyvinyl Chloride pipe technical and installation data are given in a new booklet available from Tube Turns Plastics, Inc., 2929 Magazine St., Louisville 11, Ky.

•

Vinyl Plastic over Fortisan 36 produces a fabric resistant to dust and fumes which transmits light. Called Herculite F36 Clear by Herculite Protective Fabrics, 140 Little St., Belleville 9, N. J. it is waterproof, fire and mildew resistant.

•

Fluoroflex TS, pipe and fittings lined with a high density Teflon compound which is chemically inert at temperatures up to 500 F are available now from Resistoflex Corp., Roseland, N. J. The pipe and fittings are designed to handle hot acids, including hydrofluoric and aqua regia and caustics. They are available now in standard sizes up to 4-inch with 6-inch to be available shortly.

•

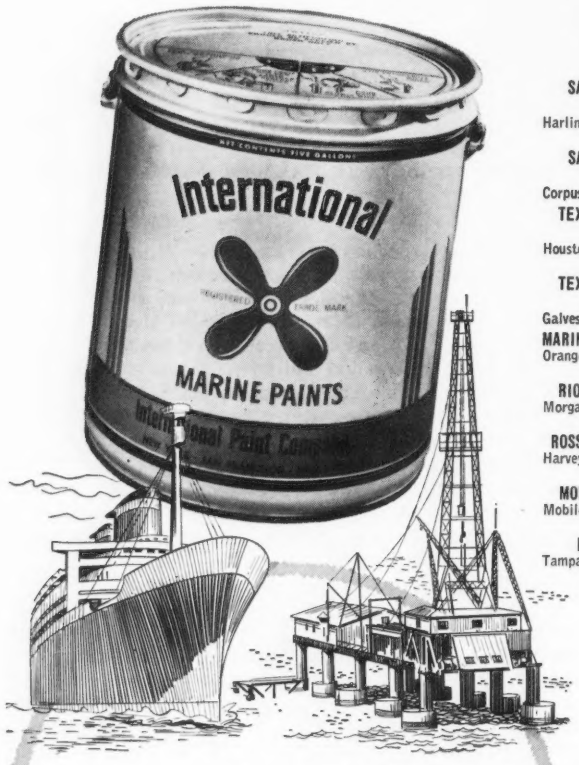
Natural or Synthetic Rubber may be bonded securely to metal by a new adhesive developed by Adhesives Dept., Raybestos-Manhattan, Inc., Bridgeport 2, Conn. Manufacturers say Ray-Bond R-86001 cures at room temperature in 24 hours or in four hours at 120-140 F. When rubber is etched before application bond is so strong rubber will tear before adhesive will part.

•

Teflon boots and sleeves available from A & A Mfg. Co. Inc., 712 South 12th St., Milwaukee 4, Wis. withstand temperatures up to 500 F.

•

Chromyl Chloride produced under a patented process by Mutual Chromium Chemical research specialists has the faculty of making textile and other surfaces water and oil repellent. It is stable and non-corrosive to most metals as long as light and moisture are excluded. Samples and technical data sheets are available from Mutual Chemicals, Allied Chemical & Dye Corp., Baltimore 31, Md.



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SAN ANTONIO MACHINE & SUPPLY CO.
Corpus Christi, Texas-Phone: 2-6591

TEXAS MARINE & INDUSTRIAL SUPPLY CO.
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Phone: WAlnut 6-1771

TEXAS MARINE & INDUSTRIAL SUPPLY CO.
Galveston, Texas-----Phone: 5-8311

MARINE & PETROLEUM SUPPLY CO.
Orange, Texas
Phone: 8-4323—8-4324

RIO FUEL & SUPPLY CO., INC.
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Phone: 5033—3811

ROSS-WADICK SUPPLY COMPANY
Harvey, Louisiana
Phone: FIllmore 1-3433

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MEN in the NEWS

Raymond L. Copson has been appointed assistant to the director of development, Solvay Process Division, Allied Chemical and Dye Corp. He has been in charge of research and development for Mutual Chemical at Baltimore and will continue to direct research and development in connection with chromium chemicals. He will be located at Syracuse.

•

John D. Keane, formerly in charge of chemical engineering research for Armour Research Foundation has accepted the post of director of research and executive secretary, Steel Structures Painting Council, Pittsburgh.

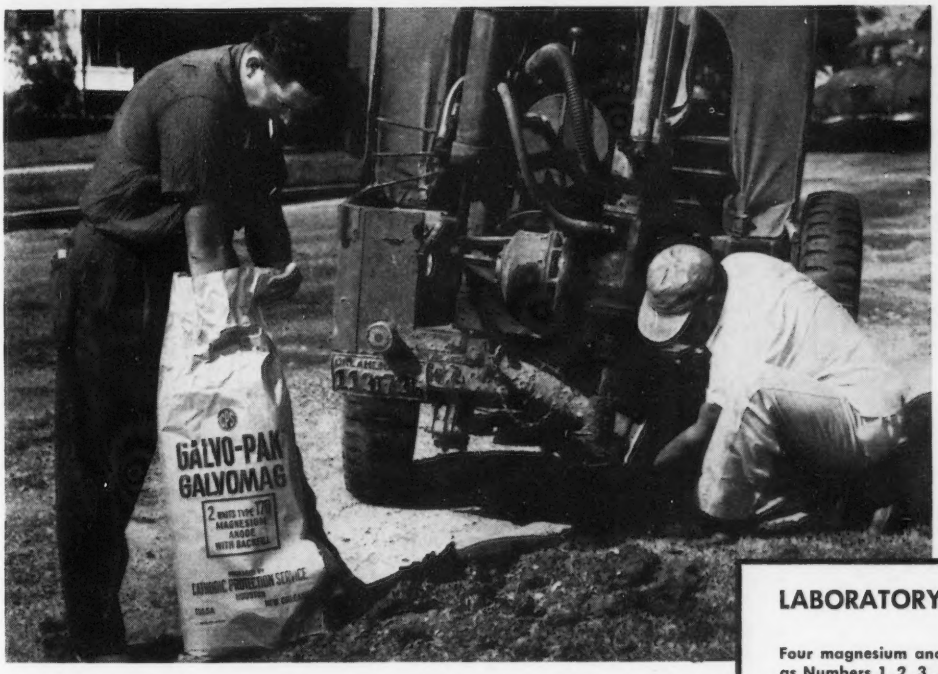
•

Jack Harris, formerly a member of the NACE board of directors and for many years active in the association has been made assistant manager—refinery and chemical valve sales, Rockwell Manufacturing Co., Pittsburgh.

•

Gordon A. Rose, has been made sales representative of Tube Turns of Can-

(Continued on Page 114)



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SAMPLE DESIGNATION	CURRENT EFFICIENCY AMP. HOURS/POUND
Number 1 (Galvomag)	539
Number 2 (Galvomag)	534
Number 3 "Equivalent" product	57
Number 4 "Equivalent" product	59

The current efficiency data obtained on Samples 3 and 4 indicates that their life will be only one-tenth the life of GALVOMAG anodes for equivalent current output. Put another way, one should pay only 10% of GALVOMAG cost for anodes equivalent to samples 3 and 4—not just 10% less.

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Seven major corrosion coordinating committees have affiliated with NACE.

•

NACE Technical task groups are formed to reach limited objectives.

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Corrosion Engineer. Needed for laboratory and field studies of corrosion problem in the Gaseous Diffusion Industry. Studies will include effects of a number of environments on existing and projected new designs. Send resume to Goodyear Atomic Corporation, Employment Department C, P. O. Box 628, Portsmouth, Ohio.

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Corrosion Engineer with five and one-half years' experience in the design, application and supervision of corrosion program on pipe lines and city distribution system. Interested in making move. CORROSION, Box 57-14.

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Certificates of membership in the National Association of Corrosion Engineers will be issued on request at $2 each, remittance in advance. The certificates, which measure 5½ x 8½ inches, are signed by the president and executive secretary of the association.

CERTIFICATES for PAST CHAIRMEN of REGIONS and SECTIONS

Certificates measuring 9 x 12 inches in size, prepared from an engraved plate, are available for issuance to regional and sectional chairmen. They will be supplied on request of the region or section at $7.50 each to be paid by the region or section, the cost to be classified as a non-reimbursable expenditure.

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NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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MEN in the NEWS

(Continued From Page 112)

ada, Ltd. with offices at 645 Lindsay Ave., St. Laurent, Quebec.

•

Herman G. Ebner has been awarded an American Viscose Corporation fellowship at Carnegie Institute of Technology for the study of chemistry, 1957-58.

•

R. H. Robinson has been named sales engineer and manager of the Buffalo district office, General Alloys, Co., 1006 Prudential Bldg., Buffalo 2, N. Y.

•

F. M. Fahrenwald, Fahralloy Co., Harvey, Ill. has been elected president of the Alloy Casting Institute.

•

L. T. Letsinger will become works manager of Aluminum Company of America's Davenport, Iowa fabricating operations.

•

Kurt Weil has been made professor and head of the Department of Mechanical Engineering at Stevens Institute of Technology.

•

R. U. Blaser, Babcock & Wilcox Co., Alliance, Ohio and **J. J. Owens,** Thompson Products, Inc., Cleveland have been given the 27th Charles B. Dudley Medal by American Society for Testing Materials for their paper "Special Corrosion Study of Carbon Low-Alloy Steels."

•

Carl G. Danielson has been named manager of the Houston Branch, Midwestern Pipe Line Products Company.

•

Fred Goldsby has been appointed assistant to the president of Chicago Bridge & Iron Co., Chicago.

•

S. C. Hamilton, formerly district salesmanager, Houston has been made a vice-president and general sales manager, with headquarters in Chicago.

•

Theodore T. Magel, formerly with Manhattan District, has been appointed assistant to the vice-president and technical director Allegheny-Ludlum Steel Corp.

•

James B. Stokes has been named sales manager for Mobile Drilling, Inc., Indianapolis, Ind.

•

Robert M. Kearns has been appointed manager of Alloy Tube Division's engineering and planning department; **Barclay Morrison** has been made manager of product engineering and development; **James P. Kelleher** is assistant to Mr. Morrison and **Wilbur J. Comerford** and **L. Carl Oseland** are product engineers.

•

Arthur C. Ruge has been named director of research and development of Electronics and Instrumentation Division, Baldwin-Lima-Hamilton Corp. **Robert P. Lathrop** is new manager of research and development.

•

Walter C. Kerrigan, assistant to the president of International Nickel Co. of Canada, Ltd. and of International Nickel Co., Inc., died at New Brunswick, N. J. July 16 after an illness of several months. He was 65.

Robert D. Schelleng has been appointed supervisor Ferrous Casting Section, of International Nickel Company's research laboratory at Bayonne, N. J. **Istvan Karsay** and **John B. Adamec** have joined the laboratory staff as research metallurgists in the ferrous metals section. **William K. Abbott** has transferred from the ferrous castings section to the company's Development and Research Division at New York.

•

Don Henderson is now Southern Louisiana Regional sales manager for Tubular Lining Corp.

•

Joseph F. Nachman is the new manager of alloy development at the University of Denver's research institute.

•

W. D. Robertson, who managed the recent NACE project inquiring into the mechanism of sulfide stress corrosion cracking has been made a full professor of metallurgy at Yale University.

•

James T. Condry has opened Southeastern Division offices for Corrosion Rectifying Co. at 2322 Airline Highway, Kenner, La., a suburb of New Orleans. The firm also has offices at Great Bend, Kansas.

•

Robert K. Morgan will represent Reilly Tar & Chemical Corp. in its Eastern territory, working out of Tuckahoe, N. Y.

•

Milton W. Snyder and **Harry Levitt** have joined the engineering department of Water Service Laboratories, Inc.

•

Benjamin Fridge has joined Menlo Research Laboratory, San Jose, Cal. as director of engineering.

•

A. G. Anderson is new district manager for The Pfaudler Company in New Mexico, Texas, Louisiana, Mississippi, Alabama, Georgia and Tennessee.

•

Roger W. Straus, formerly chairman of the board of American Smelting & Refining Co. died July 28 at the age of 65.

•

J. H. Huester, commander, U. S. Naval Reserve, is now stationed at the Naval Air Material Center, Aeronautical Structures Laboratory, Naval Base, Philadelphia, Pa.

•

P. T. Francis has been named sales manager for Quelcor, Inc., Chester, Pa.

•

T. P. May, formerly a member of Corrosion Engineering Section, The International Nickel Co., Inc., New York has been made Technical manager of the Kure Beach-Harbor Island Testing Stations of Inco. Dr. May, a director of NACE also is chairman of the association's publication committee and has been active for many years in technical committee work.

•

Lyle L. Clark has been made manager of Fansteel Metallurgical Corporation's $6.5 millions tantalum-columbium plant now under construction near Muskogee, Okla.

•

William K. Murray, has been appointed manager of technical service for Enthone, Inc., New Haven, Conn.

Remittances must accompany all orders the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

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1957 YEARBOOK
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1061 M & M Building, Houston 2, Texas
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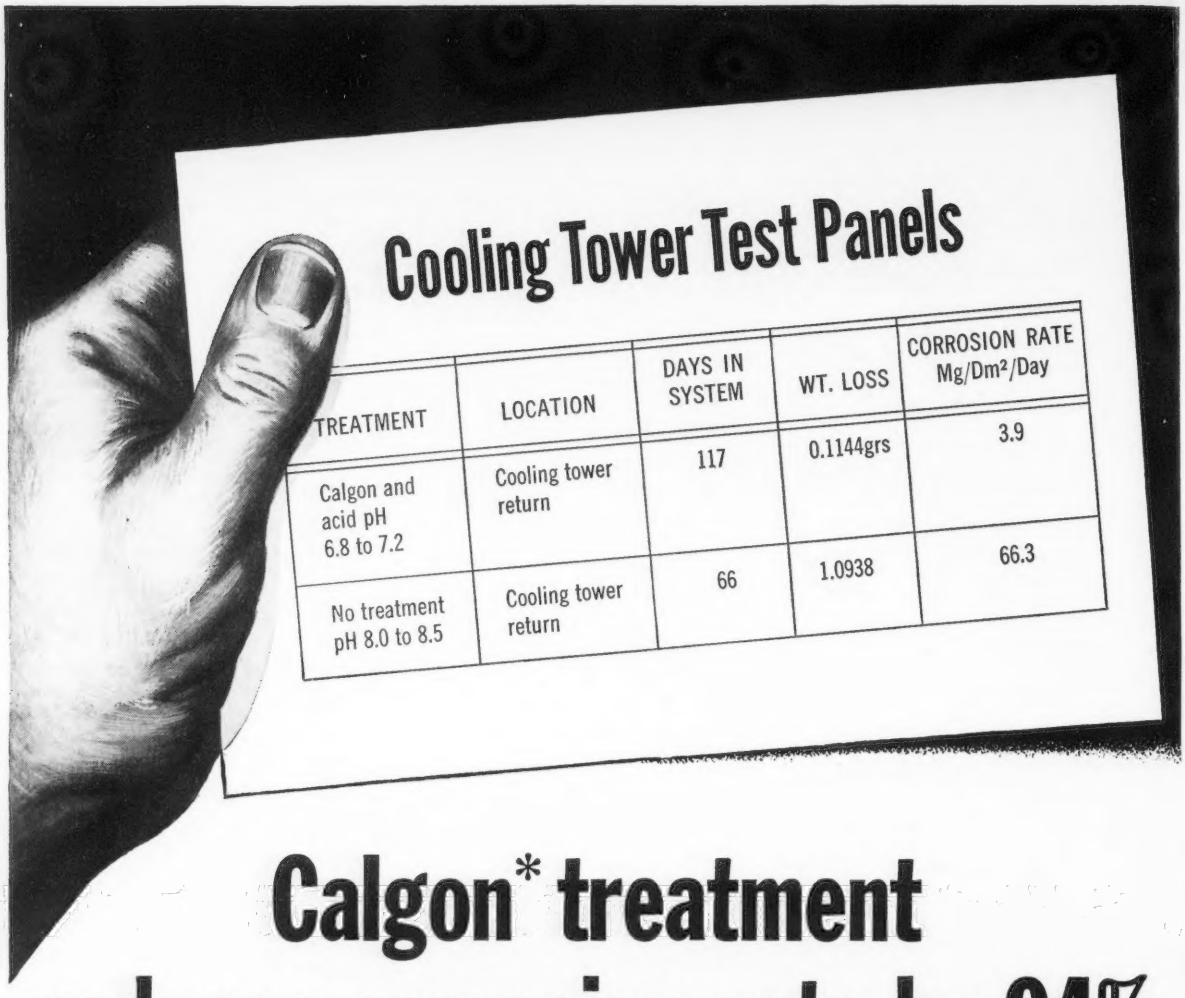

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Strength and Service Characteristics of Glass Reinforced Epoxy Oilfield Pipe*

Abstract

Important considerations in evaluating the characteristics of reinforced plastic pipe are reviewed. Fifteen tests used by the writer's company in checking strength and reliability of its product are listed. Resistance of epoxy-glass reinforced plastic pipe versus 26 acids, four alkalis, four gases, 15 salts, 11 solvents and 10 industrial fluids is tabulated. Some data are given on experience, particularly in oilfield service.

A NEW STRUCTURAL material always requires new engineering approaches. Thermosetting resins reinforced with glass fibers have been represented as simple materials. The relationship between the stress-strain of the glass cage and the resin which surrounds it, is however, complex and not simple. It is clear to many that the ultimate strength of reinforced plastic pipe and of many structural shapes of like composition is represented by the glass in the structure. That is, in a quick destruction or burst test, the strength indicated is that of the total glass structure. Likewise, the operating level at which a pipe or structure may be operated over a long period is more closely related to the resin used.

Adhesion of the resin to the fiber structure is of first importance. If adhesion is good, the ability of the resin to stretch under load while it distributes stresses to the fiber cage is next in importance. Resins are less elastic than glass, fiiberglass having an elasticity of approximately 3%, whereas, most thermosetting resins range from 1.5% to the 1.8%. One of the characteristic failures in a reinforced plastic is not failure by destruction of the glass structure, but a rupture of the resin in the structure between the glass fibers, actually a cohesive failure. In pipe this is evidenced by audible fracturing of the resin followed by weeping at high pressures. This discussion is limited to fracture weeping and not to weeping caused by porosity due to entrained air and gas.

At high temperatures, the lower tensile strength of the resin and its greater elasticity replace the fracture mechanism as a primary consideration. A yield point of reinforced plastic structures can be described as the point at which the brittle resin will fracture under stress, or, the point at which stress will develop a continuous creep along with permanent set. Creep and set is caused by overstressing the resin while it has reduced tensile strengths, such as occurs at high temperatures. This ultimately will produce failure while the fracture failure occurs quickly. Much attention has been directed to quick ultimate strengths but fracturing is often overlooked and creep history is missed entirely, due to quick timing of ultimate destruction tests.

Tests to measure the deformation or strain on a pipe under a given stress, may, when the element of time is added, measure continuous growth in deformation which is known as creep. Common steel generally is considered safe to operate with 0.2% to 0.3% strain over long periods. A reinforced plastic structure has similar characteristics. Operating stress levels on all of the writer's grades of pipe will produce no more than 0.15% to 0.28% strain, at which level there is no measurable increase with a given stress and temperature over extended times. Field data over years now confirm this. Long term pipe certification test levels are at a stress of more than twice the recommended, without creep thereby producing more than a 2 to 1 safety factor related to yield data rather than quick ultimate measurements.

The following quality testing and evaluation control procedures are used by the writer's company:

1. Short term hoop tension, stress strain tests of short duration. This test is used to check variability in the glass structure, certifying new braid designs and as a daily quality control measure. Results are related to cage strengths but this also produces fracture yield values for daily variability control.

2. Hoop tension creep tests add the element of time. These data are indicative within a 500 to 1000-hour range. Cut samples are constantly under test to check variations. These data relate to resin in the structure.

3. Surge testing is a certification test to which all pipe is subjected. One million cycles are given to the pipe at the recommended operating pressures. Surges are a violent 20 cycles per minute, with pressure differentials as high as 7 to 1. After a surge test of 1,000,000 cycles the pipe undergoes hoop tension stress strain studies and no deterioration must be indicated. This relates to adhesion, impregnation and fatigue factors of the resin.

4. The fourth test involves axial tension stress strain tests while the pipe is under pressure.

5. A fifth test adds the element of time to axial tension testing to check for creep. Here again, 500 to 1000 hours are generally sufficient to set the pattern for the product. No creep must be observed, after the initial strain on the pipe under given stress and temperature.

6. When tubing hangs down-hole 4000 to 6000 feet in wells it is subjected to external pressure, among other stresses, so short and long term collapse testing is conducted. To detect a creeping collapse a test apparatus indicates through volume changes any reduction of the inside due to external pressure. This permits selection of rated operating collapse pressures that do not produce creeping collapse. Field results over two years have proved the value of this and

(Continued on Page 118)

★ A paper presented under the title "Bringing You Up to Date on Reinforced Plastic Pipe," by H. D. Boggs, Fibercast Corp., Sand Springs, Okla, at a meeting of Western Region, National Association of Corrosion Engineers, Long Beach, Cal., November 15-16, 1956.

TABLE 1—Comparison of Working Characteristics of Commonly Used 2-Inch Plastic Pipe

Criteria	TYPES OF PIPE					
	Epoxy Glass (60%) 1000 Lb.	Polyester Glass (60%) 250 Lb.	PVC Type Type I Sched. 80	PVC Type Type II Sched. 80	Styrene Rubber Blend Sched. 80	Polyethylene Sched. 80
Recommended Long Term Maximum Working Hoop Tensile @ 80 F, Psi	13,000	5,000	1,200	1,000	750	450
Recommended Long Term Maximum Working Hoop Tensile @ Maximum Temperature—Psi	7,000	3,000	700	600	300	250
Recommended Maximum Working Temperature, Degrees F	220	130	140	140	130	125
Approximate Cost per Pound	1.80	1.10	1.50	1.50	1.20–1.55	1.00–1.40
Approximate Cost per Foot	2.15	1.10	1.50	1.50	0.80	0.80

Strength and Service—

(Continued From Page 117)

further confirm testing concepts based on time, temperature and stress factors.

7. Chemical resistance testing is as follows:

Seven-diameter length bottles of standard pipe are stress strained below the yield point and charted for each test piece. Each bottle is then weighed and measured, one end sealed off and a corrodent stored in the bottle. Bottles are kept for six months to 2 years in a heated oven and a duplicate is kept at room temperature. After a given test period the corrodents are poured out, the bottle is unsealed, reweighed and then re-stress strained and compared to the original in order to determine if any change in the strength of the pipe has taken place. This information directly indicates what might be expected when the pipe is installed in service, whereas, cut pieces are not indicative and give no strength values before or after the test.

8. Measuring the coefficient of expansion of the pipe is a standard procedure, using a thermocouple to measure temperature rise and a dial indicator to measure growth in a 21-foot length.

9. The coefficient of heat transfer also is a standard test using calorinic measurements to indicate heat transfer in a given time.

10. Beam modulus testing is a 3-point loading test in which the data are converted to the beam modulus formula.

11. All pipe undergoes production testing for five minutes at 150 percent of the recommended operating pressure. Visual inspection is made before and after the test. A principal consideration is tightness of the threads in sealing against the pressure.

12. All fittings are gauged for size and pressure tested at 150 percent the recommended operating pressure for five minutes.

13. During the fabrication the braid undergoes inspection, gauging for size, checking for braid angle and is inspected visually for quality.

14. Raw materials are quality checked as received, by Gelometer tests and other laboratory procedures to determine quality of hardeners and the resins. When necessary, cast bars are made and stress-strain studied to check resin and hardener quality.

15. A very important test is for edgewise porosity. A 2½-inch ring of the pipe is subjected to water on its cut edge at pressures as great as 1000 psi for 500 to 1000 hours to determine if there is edge porosity. Edgewise porosity is unacceptable because a structural reinforced plastic which is porous edgewise will give trouble under cyclic stress in wet environments.

Characteristics of Reinforced Plastic Pipe

A braided glass structure can be designed for a given structural load. For instance, axial strength is increased for uses where it hangs under stress loads in a disposal well, or hoop strength is increased for use in applications involving high internal pressure. Its structure can be changed to make it more suitable for beam loads.

All resin in pipe made for the writer's company is cast integral with the glass structure. A resin-rich interior is provided which has better chemical resistance to the fluids in the pipe and results in a factor of C 150.

The thread system is built around the American Standard 60 Degree Stub System. It is a full upset thread on a ⅛-inch to 1-foot taper. Primary advantages are that it does not destroy the reinforcement in the pipe, develops shear stresses beyond that needed for 100% joint strengths, will not cross-thread, is sturdy and dependable, resisting chipping and damage in transit and is easy to duplicate in molds to assure matched reproduction in molding.

In more than five years since plastic pipe production began in 1951 some

(Continued on Page 120)

TABLE 2—Chemical Resistance of Commonly Used Plastic Pipe

Types of Pipe	Epoxy Glass (60%)		Polyester Glass (60%)		PVC Type I		PVC Type II		Styrene Rubber Blend		Polyethylene	
Temperature Degrees Fahrenheit	80 F	180 F	80 F	130 F	80 F	140 F	80 F	140 F	80 F	130 F	80 F	125 F
ACIDS												
Acetic 10%	E	E	E	E	E	E	E	E	E	E	E	E
Acetic, Glacial	E	X	E	F	E	U	F	U	U	U	U	U
Benzene Sulfonic 10%	E	E	E	E	E	E	E	E	E	E	E	E
Benzoic	E	E	E	E	E	E	E	E	E	E	E	E
Boric	E	E	E	E	E	E	E	E	E	E	E	E
Butyric	E	F	E	F	F	U	U	U	U	U	U	U
Chloroacetic 10%	E	E	E	E	E	E	E	F	U	U	U	U
Chromic 5%	F	U	E	U	E	E	E	E	E	U	E	E
Citric	E	E	E	E	E	E	E	E	E	E	E	E
Fatty Acids	E	X	E	E	E	U	E	U	E	F	F	U
Formic	E	F	E	F	E	U	E	U	U	U	E	F
Hydrochloric	E	E	E	E	E	E	E	E	E	F	E	E
Hydrobromic	E	E	E	E	E	E	E	E	E	F	F	E
Hydrocyanic	E	E	E	E	E	E	E	E	E	E	E	E
Hydrofluoric	X	X	U	U	E	E	E	E	E	F	E	E
Hyprochlorous	E	X	E	F	E	E	E	E	E	F	E	E
Latic	E	E	E	E	E	E	E	E	E	F	E	E
Maleic 25%	E	E	E	E	E	F	E	F	E	E	E	E
Nitric 5%	E	F	E	F	E	E	E	E	E	U	E	E
Oleic	E	E	E	E	E	F	E	U	E	F	U	U
Oxalic	E	E	E	E	E	E	E	E	E	E	E	E
Perchloric	E	E	E	E	E	E	E	E	E	F	E	E
Phosphoric	E	E	E	E	E	E	E	E	E	F	E	E
Picric	E	E	E	E	U	U	U	U	E	E	E	E
Stearic	E	E	E	E	E	E	E	U	E	F	U	U
Sulfuric 50%	E	E	E	E	E	E	E	E	E	E	E	E
Alkalies												
Ammonium Hydroxide	E	F	E	F	E	E	E	E	E	F	E	E
Calcium Hydroxide	X	X	U	U	E	E	E	E	E	F	E	E
Potassium Hydroxide	X	X	U	U	E	E	E	E	E	F	E	E
Sodium Hydroxide	E	X	U	U	E	E	E	E	E	F	E	E
Gases												
Chlorine—Dry	E	F	E	F	E	E	E	E	E	F	U	U
Chlorine—Wet	F	U	F	U	E	E	E	E	E	U	U	U
Sulfur Dioxide—Dry	E	E	E	E	E	E	E	E	E	E	E	E
Sulfur Dioxide—Wet	E	E	E	E	E	E	E	E	E	E	E	E
Salts												
Aluminum Sulfate	E	E	E	E	E	E	E	E	E	E	E	E
Ammonium-Chloride, Nitrate & Sulfate	E	E	E	E	E	E	E	E	E	E	E	E
Copper-Chloride, Nitrate & Sulfate	E	E	E	E	E	E	E	E	E	E	E	E
Ferric-Chloride, Nitrate & Sulfate	E	E	E	E	E	E	E	E	E	E	E	E
Nickel-Chloride, Nitrate & Sulfate	E	E	E	E	E	E	E	E	E	E	E	E
Stannic Chloride	E	U	E	E	E	E	E	E	E	E	E	E
Zinc Chloride	E	E	E	E	E	E	E	E	E	E	E	E
Boron Sulfide	E	F	E	F	E	E	E	E	E	E	E	E
Sodium Bicarbonate	E	E	E	E	E	E	E	E	E	E	E	E
Sodium Carbonate	E	E	E	E	E	E	E	E	E	E	E	E
Neutral Salts	E	E	E	E	E	E	E	E	E	E	E	E
Solvents												
Acetone	E	U	U	U	U	U	U	U	U	U	U	U
Alcohol	E	E	E	E	E	E	E	F	E	U	E	U
Aniline	F	X	U	U	U	U	U	U	U	U	U	U
Benzene	E	X	E	U	U	U	U	U	U	U	U	U
Carbon Tetrachloride	E	F	E	F	F	U	U	U	U	U	U	U
Chloroform	E	F	E	F	U	U	U	U	U	U	U	U
Ethyl Acetate	E	E	E	U	U	U	U	U	U	U	U	U
Ethylene Chloride	E	F	E	F	U	U	U	U	U	U	U	U
Formaldehyde	E	E	E	E	E	E	E	E	E	E	E	E
Phenol	E	F	E	F	E	E	E	F	E	F	E	E
Trichloroethylene	E	F	F	U	U	U	U	U	U	U	U	U
Industrial Fluids												
Black Liquor	X	X	U	U	E	E	E	E	E	F	E	E
Sulfite Liquor	E	E	E	E	E	E	E	E	E	E	E	E
Bleach	E	F	E	F	E	E	E	E	E	U	E	E
Photographic Developers	E	E	E	E	E	E	E	F	E	E	E	E
Pickling Acid-Sulfuric & Hydrochloric	E	E	E	E	E	E	E	E	E	E	E	E
Plating	E	E	E	E	E	E	E	E	E	E	E	F
Petroleum	E	E	E	E	E	E	E	E	E	E	U	U
Tanning	E	E	E	E	E	E	E	E	E	E	E	E
Soap	E	E	E	E	E	E	E	E	E	F	F	U
Sewer	E	E	E	E	E	E	E	E	E	E	E	E

Legend: E—Excellent—No Question.
F—Moderate attack but satisfactory.
X—Has many economic applications. Test study required.
U—Unsatisfactory.

PVC PIPING NEWS

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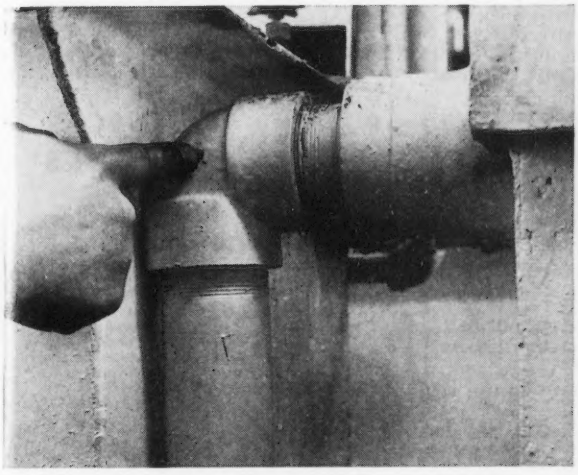

DRAIN LINES NOW PERMANENT. Former steel drain pipe and fittings on this pickle rinse tank oxidized quickly . . . lasted about a month. Replacement with PVC pipe and **ttp** fittings stopped this costly maintenance. Line is 2", schedule 80. Courtesy North Electric Company, Galion, Ohio. Contractor: Charles Hoffman Co., Mansfield, Ohio.

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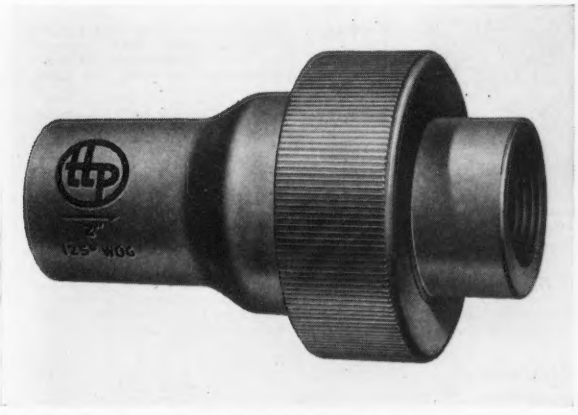

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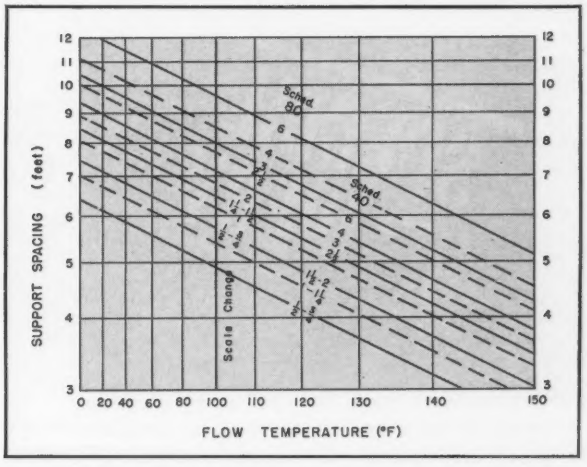

HOW TO SUPPORT PVC PIPE. This chart gives recommended support spacing for PVC pipe of different schedules and sizes. Full explanation of this procedure and other valuable application data are given in booklet TTP 119R, free on request.

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Strength and Service—

(Continued From Page 118)

800,000 feet of reinforced plastic pipe has been put in service. New epoxy pipe has a record of three years without a single field failure, even though it is operating at high pressures where steel, plastic and cement-coated steel, protected steel, stainless steel and other materials often last as little as three months. Some field service records show original pipe in use as long as five years and still in service. Pipe made earlier of polyester resins and mat had failures, however, which have been eliminated with epoxy pipe. Sections of the epoxy pipe tested for comparison with the original produced strength after six months' to 2 years' exposure have changed less than 3 percent from the norm and in appearance are as if new. Strength figures related to epoxy pipe and the particular types of epoxy resins and braided glass reinforcements used by the writer's company are:

1. Hoop strengths at burst, range from 20,000 to 42,000 psi dependent on glass structure.

2. Yield strengths range from 13,000 to 20,000 psi.

3. Hoop stress at the production test levels range from 5600 to 13,000 psi.

4. Hoop stress at recommended operating levels range from 3700 to 9300 psi for room temperature operations, 3750 psi to 4700 psi for temperatures up to 180 F and to 220 F for heat grades. All are expected to give more than a 5-year life under adverse conditions.

5. Maximum axial tension at hoop burst ranges from 14,000 to 28,250 psi.

6. Axial tensions yield from 6500 to 10,000 psi. Tubing which hangs downhole in wells to 6000 feet has axial strength ranges from 16,000 to 43,000 lb. ultimate, depending on size and grade.

7. Ultimate shear strengths of the threaded joint on the 2⅜-inch pipe is 32,800; on 2⅞-inch pipe, 48,200 and for the 3½-inch pipe 68,000 lb. Joint strength is from 150 percent to 200 percent that of axial strength of the pipe.

8. Hazen-Williams flow factor is 150.

9. The coefficient of expansion varies according to its construction but it ranges from 7.06×10^6 to 8.25×10^6 in/in/° F. This is slightly more than steel.

10. Beam modulus rating for two 3½-inch grades: 0.19 inch wall pipe runs 21,700 and the 0.26-inch wall, 26,400 psi.

11. Weight is less than one-fourth that of steel and ranges as follows:

2⅜-inch pipe weighs 0.63 to 1.5 lb. per foot. Operating pressures range from 500 to 1000 psi.

2⅞-inch pipe weighs from 0.8 to 1.45 lb. per foot and the operating pressures from 450 psi to 1000 psi.

3½-inch pipe weighs from 1.07 to 2.07 lb. per foot and the operating pressures from 350 psi to 1000 psi.

All weights include threads and coupling on the joint.

Strengths of this material place it in the range of metal and well above those of extruded thermoplastics. Operating hoop stress levels recommend for thermoplastic pipe range from 500 to a maximum of 1000 psi with no safety factors allowed for creep history. Reinforced epoxy pipe strength is 6 to 10 times that of thermoplastics and all stress levels will take surge loads which are dangerous to thermoplastics. Operating temperatures as high as 220 F are recommended and in field trials epoxy pipe has been in service at temperatures up to 300 F with no failures.

McFarland to Talk

Dr. Kenneth McFarland, educational consultant to General Motors Corp. will speak at the annual South Central Region business luncheon. It will be held Thursday, October 3 at noon in the Persian Room, Skirvin Tower Hotel.

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T-6A Report on Epoxy Resins. A Report of NACE Technical Unit Committee T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion. Publication 56-5. Per Copy $.50.

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TP-6G First Interim Report on Recommended Practices for Surface Preparation of Steel. (Pub. 50-5). Per Copy, $.50.

TP-6G Second Interim Report on Surface Preparation of Steels for Organic and Other Coatings. (Pub. 53-1.) Per Copy, $1; five or more copies to one address, per copy $.50.

T-6K Acid Proof-Vessel Construction with Membrane and Brick Linings. A Report of NACE Technical Unit Committee T-6K on Corrosion Resistant Construction with Masonry and Allied Materials. Publication No. 57-6. Per copy $.50.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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Field experience has proved that Corban extends the life of tubing, casing, fittings, and valves. It also saves time and production by helping avoid workovers necessitated by corrosion-damaged equipment.

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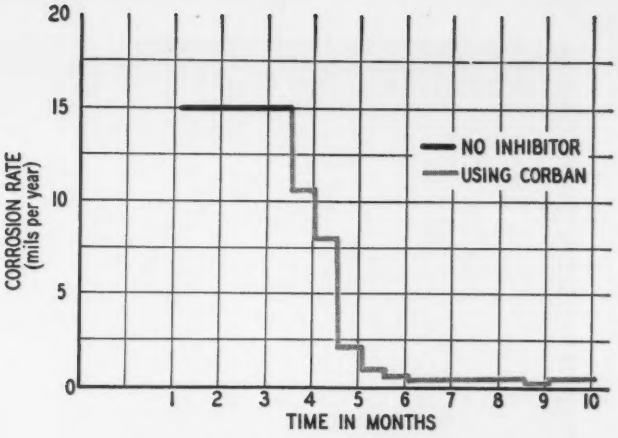
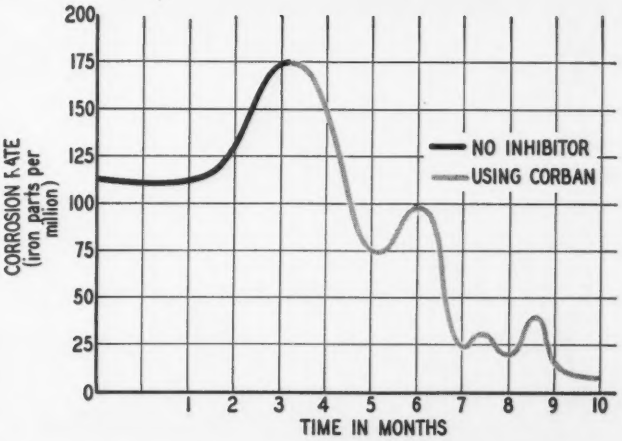


1 STANDARD TEST uses metal strips inserted in wellhead or tubing. Surface penetration or weight loss indicates degree of corrosion.



2 FLUID ANALYSIS is quick test of corrosion in gas condensate wells. Chart shows parts per million of dissolved iron in water sample.

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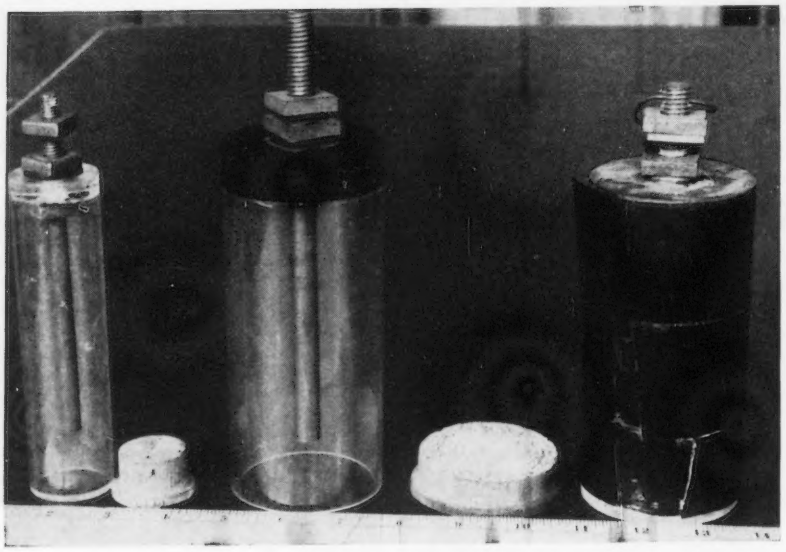

Figure 1—Left to right: Showing components of original size of half-cells. Middle, 2¾ inch size before filling and activation and right, completed half-cell wrapped with tape to exclude light.

A Simplified Method for Making And Maintaining Half-Cells*

Abstract

A method is described for making inexpensive copper-copper sulfate half cells that compare favorably in characteristics with recommended behavior of half cells in technical literature. Routine handling of these cells is described.

THE COPPER-COPPER sulfate half cell is used almost exclusively in measuring the potential of metallic structures with respect to the soil or water with which they are surrounded. Many versions of this cell are on the market or have been made by various users. This article proposes to describe a method of making and handling half cells.

The simplest half cell consists of a piece of electrolytic copper rod, a short length of plastic tubing filled with a saturated solution of copper sulfate and closed with a wooden plug. Half cells described herein are essentially like this, but the method of caring for them may differ somewhat from usual practice.

In preparing a half cell for use, the plastic tube, one end closed and containing the copper electrode is placed open end up in a beaker containing a solution of nitric acid (made by diluting concentrated acid with an equal volume of water) which covers the copper.

After a minute or two, the acid is poured out and the tube and copper rinsed with clean water several times. Then enough copper sulfate crystals are added to fill at least half the plastic tube, followed by enough distilled water to cover the copper electrode.

A loosely fitted dry wooden plug is inserted in the open end of the cell, the half cell is turned over and placed wooden plug side down into a beaker of distilled water.

After an hour or so the wooden plug has swelled enough so that the half cell may be handled or used. Before use, however, the half cell is wrapped in black plastic tape to exclude light. Figure 1 shows two half cells before filling and one filled, wrapped and ready for service.

No half cell is used more than 60 to 90 days, after which it goes back to the laboratory (or shop) for replacement. If it shows any defects such as leaking or change in color, it goes back sooner. While in use the half cell sits, wooden plug down, in a glass or plastic container containing water except when measurements with it actually are being made.

When it goes back to the laboratory it is placed upside down (wooden plug up) in a corner. After a week or so the wooden plug dries out and can be removed, the solution can be dumped out and the half cell goes into dry storage.

The labor required to handle the half cell in this manner is so small that any time a field crew has any doubts about half cells they are encouraged to send them in for replacement.

Half cells prepared in this manner have been checked many times using a Leeds and Northrup or Beckman saturated calomel half cell. (The cell formed by dipping a calomel half cell and a copper-copper sulfate half cell into a beaker of Louisville City water (Ohio River water, resistance about 2000 ohms/cu.cm.) has a potential of about 70 millivolts varying from 68 to 73.) This is exactly the behavior of the most carefully made half cells described in the technical literature.

Most half cells returned from a tour of duty in the field give readings in the same range, when coupled to a calomel half cell, as the freshly-prepared cells.

This routine with half cells has been in use for about five years. The first group of half cells made up in the manner were six inches long and one and one-half inches in diameter. Their only fault was that they would sometimes lay down on the job unless held in place.

When made with a 2¾ inch Plexiglass tubing they stand up without being held.

★ A paper presented under the title "The Care and Feeding of Half-Cells," by Lewis P. Aker, Louisville Gas & Electric Co., Louisville, Ky. at a meeting of Northeast Region, National Association of Corrosion Engineers, Niagara Falls, N. Y., October 31-November 3, 1955.

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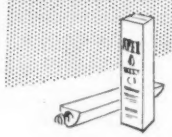

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TOPICS NEXT MONTH

Part 2—Use of Stainless Steel to Combat Corrosion in the Chemical Industry

Controlling External Corrosion on the Trans-Arabian Pipe Line

Capitelized Coating Costs Method Economical for Southeastern Pulp and Paper Mill Steel

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makes this a model pipe wrapping plant

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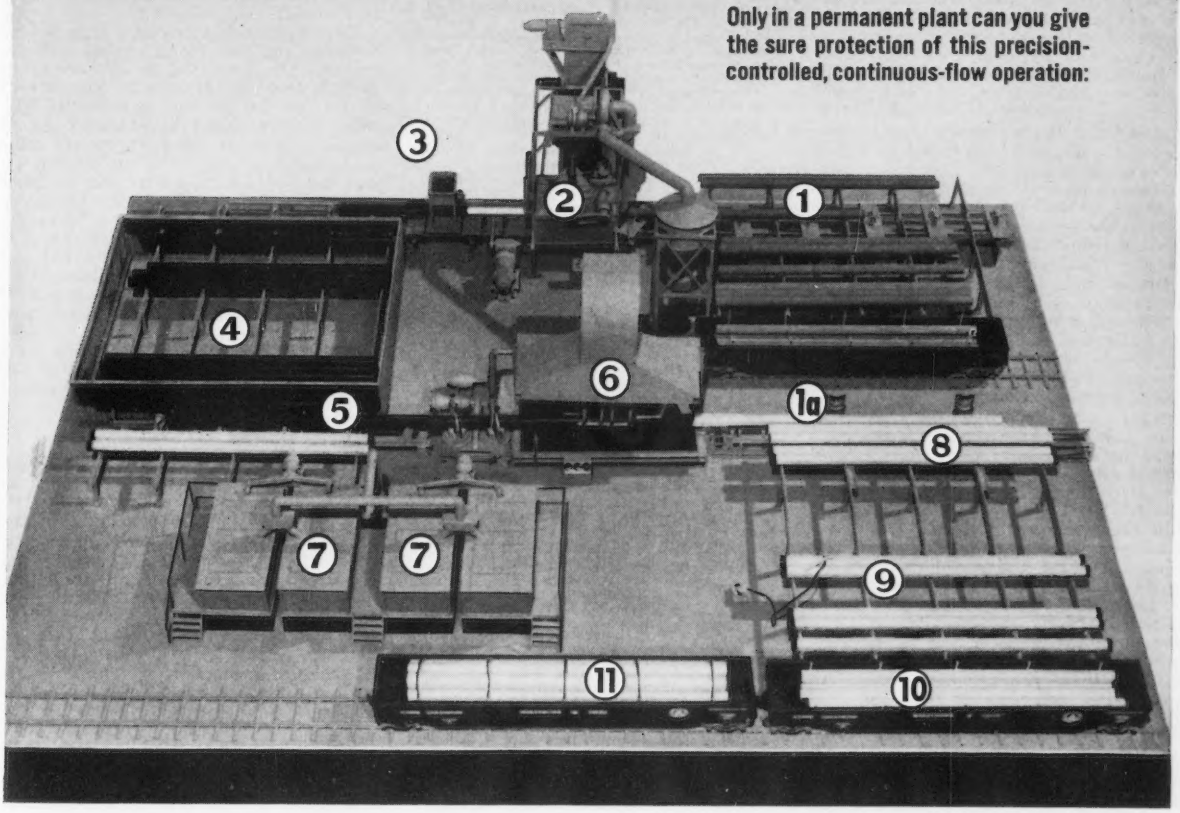

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1a. Bare pipe is unloaded direct from car in plant.
2. Roto-grit blaster removes all trace of rust, mill scale.
3. Machine applies primer.
4. Primer cures in heat room.
5. Automatic conveyor delivers pipe to—
6. Coating and wrapping machine.
7. Automatic kettles flow enamels to coating and wrapping machine.
8. Coated and wrapped pipe is delivered to an electronic-inspection area.
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Part 1 of 2 Parts

Use of Stainless Steel to Combat Corrosion In the Chemical Industry*

Abstract

After discussing briefly reasons for the corrosion resistance of stainless steels and some of the environments in which it corrodes readily, the author describes maintenance and structural errors which can cause rapid corrosion. Some errors in the customary concept of oxidizing and reducing conditions are noted. In a discussion of specific types for specific services an understanding of the meaning of specifications is urged, with the observation that variations in process streams are more significant than minor alloy variations. Some errors in assessing the meaning of quality control tests are enumerated and relative values of tests and metallographic examinations considered. Behavior, sometimes unexpected, of some alloys in screening tests is considered.

In a discussion of welded specimens reasons for not using them in quality control tests are enumerated. A specification for drawn welded tubes in services commonly associated with seamless tubing is described.

Behavior of some steels is described in acetic acid, sulfuric acid, caustic, sulfurous acid, phosphoric acid and nitric acid environments. Reasons for using stainless in some cooling waters are given, and dangers associated with their use explored.

Characteristic defects of stainless materials are considered with special emphasis on sensitization and stress-corrosion cracking. The author considers concepts of emphasis on sensitization overemphasized and believes stress-corrosion cracking to be more common than is indicated in literature. Illustrations of stress-corrosion cracking are given.

Recommendations on fabrication include proper selection of rod analysis, quench phenomena and welding repairs.

Some comments are included on corrosion testing, including size of specimens and apparatus for inserting in process streams.

The Nature Of Stainless Steels

THE OUTSTANDING characteristics of all stainless steels is their unexpected resistance to corrosion in many environments as compared with nonchromium bearing ferrous alloys. This corrosion resistance, which becomes startlingly apparent at about 12 percent chromium is the hall-mark of the stainless steels, and has been called by Evans a "spurious nobility."[1]

This corrosion resistance usually is referred to as passivity. According to Uhlig[2], the passivity of stainless falls under his definition No. 1, "an alloy, active in the EMF series, when its electrochemical behavior becomes that of an appreciably more noble material."

The exact nature of passivity is still a matter of conjecture. Among the theories currently held are the "Generalized Film Theory" (referring to either metallic oxide films or adsorbed oxygen) and the "Electron Configuration Theory." According to Evans, the behavior of a metal is strongly related to the solubility of its oxides, which fact, he contends, supports the oxide film theory. On the other hand, the oxygen film theory, which has been put forward by several investigators, is apparently opposed by the action of chromates, nitrites and similar inhibitors. According to the electron configuration theory, iron is passivated by sharing electrons with the naturally passive chromium constituent, when the iron:chromium ratio is less than approximately 5:1. This proportion corresponds to about 15.7 percent chromium. The observed 12 percent minimum chromium figure for conventional stainless is probably due to the interaction of other alloying elements. This theory is opposed, to a certain extent, by the fact that stainless is not passive in **all** environments. According to the electron configuration theory, there is a largely monatomic or monomolecular surface film in which adsorbed material shares electrons with the surface atoms and reduces the reactivity of the surface atoms. Collins[3] has reported surface films consisting of "hydrous oxide films enriched with silica" and notes that the silicon content of the film is several times that of the metal proper.

Regardless of the exact mechanism, it is known that the film formed on stainless steel is highly protective and that it does affect the electrochemical behavior and the solution potential of the metal. The insoluble surface film tends to be impermeable to many corrosives. This is in contrast to the rusting of steel, for instance, where the rust usually is only slightly protective. It is known that the film on stainless is enhanced by oxidizing conditions, whence, the good resistance of stainless alloys to nitric acid, oxidizing agents and high temperature oxidation.

On the other hand, the so-called "reducing acids," such as dilute sulfuric or hydrochloric, attack the alloy at imperfections in the protective film. This corrosion evolves hydrogen which then causes electrochemical reduction of the protective film, finally allowing general attack of the now active metal. When the stainless steels are in their active condition, their electrochemical behavior is very similar to that of ordinary iron and steel—if not worse.

Sandblasting Feasible Sometimes

The passive surface film may be removed mechanically as well as chemically and there have been instances in which mechanical abrasion has led to serious attack, usually of a local nature. However, mechanical damage will not necessarily result in accelerated attack. For instance, it is true that sandblasting stainless steel will put the surface in an active condition. However, the metal surface is active for only as long as it takes to adsorb oxygen from the atmosphere, or from an aerobic environment, to become passive again.

In one case, a failure of Type 316 heating coils in a batch still kettle, originally was ascribed to sandblasting. In service, these coils become coated with a carbonaceous deposit due to the high surface temperatures and this must be removed at intervals. Hand-cleaning is laborious so, on one occasion, the surface tars were removed by sandblasting. When the coil was put back in service, rapid failure ensued.

The failure was ascribed at first to the sandblasting, but more serious consideration of the length of time between the sandblasting operation and the return to service led to the conclusion that other factors might be involved. Field corrosion tests on freshly activated coupons failed to duplicate the attack. It was learned subsequently that an acid catalyst had been employed inadvertently in higher concentrations than normal and had caused the corrosion. At present the author's company has no hesitancy in cleaning stainless steel equipment by sandblasting, provided several hours elapse before it goes back into service. In a batch still, it is preferable that sandblasting follow a corrosive service and immediately precede the most innocuous service for the kettle.

Galvanic Relationships Important

The activity or passivity of stainless steel is affected profoundly by galvanic relationships, depending on environment and driving potentials. Making stainless the anode in a galvanic couple tends to promote passivity in many environments, presumably because the anode is the oxidizing electrode. Making stainless a cathode promotes activity because of interstitial action by hydrogen. This, of course, is contrary to normal principles of cathodic protection, in which polarization to the open circuit potential of an anode is attempted in order to protect it from corrosion. Such treatment may result in activity of stainless steel with subsequent rapid attack. This mechanism has been suspected in one failure of a stainless-clad steel tank in sulfuric acid service.

In dilute aqueous environments, austenitic stainless steels are noble to the other common materials of construction, excepting the true noble metals. It must be remembered, however, that each different chemical environment has its own galvanic series.

Although both stainless and copper can cause severe galvanic corrosion of steel in water, under certain conditions there may be no attack. It is quite common, for instance, to put steel shells over stainless bundles with water on the shell side of a condenser in once-through cooling systems. This application is successful, however, as largely because of anaerobic conditions in polluted river water. Under such conditions the use of steel shells over copper bundles also should be satisfactory.

Some Couples Disastrous

In open recirculated systems, coupling large areas of 18-8 to steel is disastrous. For instance, with an area ratio of 40/1 (stainless-to-steel), the corrosion rate of steel is accelerated twenty-fold (.004 IPY becoming .080 IPY) even in the

(Continued on Page 126)

★A paper by Charles P. Dillon, Union Carbide Chemicals Corp., Texas City, Texas presented at a meeting of Niagara Section, National Association of Corrosion Engineers, at Buffalo, N. Y., May 9-10, 1956.

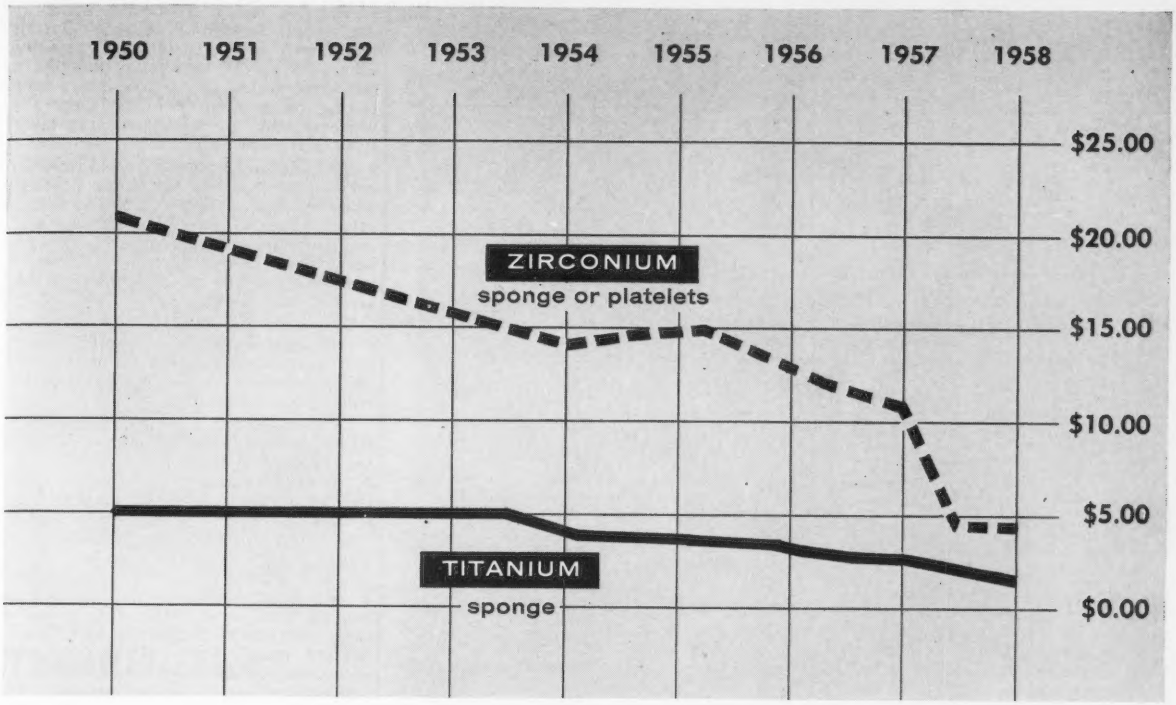

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TABLE 1—Variations in Corrosion Rate Among Metals and Alloys Commonly Used in Chemical Process Environments

MATERIAL	Acetic Acid Esterification	Acetic Anh. Distillation	Amine Distillation	Amine Distillation	MEA Recovery
Type 316	.007	.002	.005	.003	.007
Type 304	.010	.018	.310	.030	.015
Type 316-3 Cu	.023	....	....	....	....
Carpenter 20	.036	....	.009	....	....
Copper	.060	.070	.013	.003	....
Hastelloy C	.100	....	.001	.007	....
Hastelloy B	.110	....	.021	....	....
8 Percent Al. Bronze	....	.052	....	....	....
Monel	....	....	.015	.008	....
70-30 Yellow Brass	....	....	....	....	.003

TABLE 2—Composition of Experimental Heats of Type 316 Stainless Steel for Investigation of Compositional Effects on Corrosion

ELEMENT	Off-Grade ASTM	Alloy CIAB	Alloy UCC	Press. Vessel 316-L
Carbon	0.10	0.07	0.05	0.021
Manganese	1.76	1.52	1.75	1.73
Silicon	0.75	0.62	0.66	0.43
Phosphorous	0.017	0.022	0.027	0.024
Sulfur	0.017	0.010	0.009	0.016
Chromium	16.5	18.4	17.05	17.7
Nickel	10.5	10.2	12.6	13.5
Molybdenum	2.4	2.7	2.5	2.2

presence of chromate inhibitors. The corrosion rate of copper is doubled when coupled to 316 in this case (that is from .0006 IPY to .0012 IPY).

Generally, stainless steels are passive under "oxidizing" conditions and active under "reducing" conditions. This generalization stands up well as long as the oxidants are confined to nitric acid, strong sulfuric and chromate solutions and the reducing agents to hydrochloric acid (which is oxidized by strong oxidizing agents).

This concept, i.e. of oxidizing and reducing conditions, becomes unwieldy in view of the fact all conversion of metal to ions involves oxidizing reactions.

In chemical process streams it frequently is impossible to predict whether a given environment is "oxidizing" or not. Further, this is not a "go-no-go" proposition. Properly speaking, different environments have different oxidizing capacities.

This difficulty is illustrated by Table 1 in which the corrosion resistance of different metals is shown in a number of environments. It is apparent that it would have been difficult to predict an order of merit. Further, relative corrosion resistance is not indicative of galvanic relationships, because anodic and cathodic polarization effects will come into play in a galvanic couple. In practical applications in the chemical industry it is difficult, if not impossible, to predict the galvanic relationships between stainless steels and other common materials of construction unless one has studied the situation beforehand. Properly designed and executed tests are necessary if one is to arrive at a realistic solution to this problem. Frequently the transposition of data from one apparently related service to another, as in the case of once-through versus recirculated cooling water cited above, can be disastrous.

Oxidizers Not Always Beneficial

Before closing this section, note that oxidizing agents are not always beneficial. For instance, just as corrosion of steel in water is caused by oxygen, yet inhibited by large quantities of oxygen or oxidizing agents, so the corrosion of 18-8 may be accelerated by amounts of oxidizing agents insufficient to passivate. It is common knowledge, for instance, that cupric ions can inhibit the corrosion of 18-8 in certain concentrations of sulfuric acid otherwise corrosive. Yet insufficient amounts may accelerate attack (especially at crevices) and the corrosion may involve electrochemical plating of copper and rapid dissolution of the stainless steel.

Note further that the attack at locally active anodic areas on stainless steel involves electrochemical corrosion in which oxygen or oxidizing agents serve to depolarize the large cathode area and increase the pitting rate. It is true, of course, that the anodes occur at oxygen-deficient locations and that it is precisely this oxygen deficiency which accelerates pitting under deposits or in crevices. Nevertheless, it is frequently possible to minimize attack by a thorough deoxidation of the environment, especially for those mild corrodents in which this does not involve serious corrosion of active stainless steel. In other words, "in the complete absence of oxygen or other oxidizing agents, the cathodic areas will not be depolarized and the development of pits will be arrested."[1] This is not necessarily true of stress corrosion cracking, as will be noted later.

In summary then, the outstanding resistance to corrosive attack of the stainless steels is due to some sort of a surface film mechanism which is known to be promoted by oxidizing agents. On the other hand, it is essential that specific conditions be understood before predicting whether or not the presence or absence of oxidizing agents will be beneficial or may lead to accelerated localized attack.

Use of Stainless Steels In Specific Services

The largest use of martensitic alloys is in steam turbine blades and high temperature parts. In cooling water Type 410 may be serviceable, yet is prone to rapid pitting attack under stagnant conditions as shown in Figure 1. The same alloy, however, can be very serviceable under conditions which do not permit deposition of solids on the metal surface. For instance, Type 410 is successfully used as a pump impeller in the same water which will cause rapid pitting on condenser tubes. Generally, however, the use of martensitic alloys is very limited in chemical plants.

The ferritic grades are superior in corrosion resistance to Class I alloys. They are used commonly for nitric acid service and where only contamination is a problem, as with food products. Yet, because of their rather poor strength, impact resistance and weldability, they are not very popular.

The austenitic grades, however, have a rare combination of desirable properties, as explained before, and it is the 300 series alloys with which this article will be concerned primarily.

In considering the use of stainless steels for plant services, specifications are the first thing to be considered.

A. **Specifications.** Specifications for stainless steel may be exemplified by ASTM A-240-54, Corrosion Resisting Chromium and Chrominum-Nickel Steel Plate, Sheet and Strip for Fusion Welded Unfired Pressure Vessels. Such a specification covers the process of manufacture, heat treatment, composition, mechanical properties and "qualification tests." Individual companies sometime extend these specifications by calling for more restrictive compositions or more rigid requirements of mechanical and physical properties.

There is good agreement as to manufacturing processes, heat treatment (except for failure to note the significance in certain environments) and the broader compositional limits, e.g. between 304 and 316 stainless steel.

There is considerable disagreement, however, on the compositional requirements for 316, for instance. A survey by the Chemical Industry Advisory Board of the American Standards Association showed that numerous chemical companies have individual ideas on chromium, nickel, molybdenum and carbon requirements.

In the writer's opinion, the variations in process streams overshadow minor variations in alloy composition. Preliminary work has been completed on four heats of Type 316 of the compositions shown in Table 2. Observe that the alloys vary from a quality that will not pass A-240 to a"good" grade of high chromium, low carbon 316. The alloy CIAB has been deliberately thrown off-balance and has about 8-10% ferrite. Laboratory tests show wide variations in the order of merit among these heats. In field corrosion tests in the author's plant, however, in those environments where 316 is the proper material of construction, there was no significant difference in the corrosion behavior of these experimental heats. Further, it was not unusual to find off-grade A-240 material slightly better than the low carbon "pressure vessel quality" alloy.

Qualification Tests Misunderstood

A major difficulty in specifying stainless steel sometimes arises from a misunderstanding of the qualification tests. The most common of these are the Huey Test (boiling 65% nitric), the Strauss Test (acidified copper sulfate), and the Electrolytic Oxalic Acid Etch Test. Too few understand the true pur-

(Continued on Page 128)

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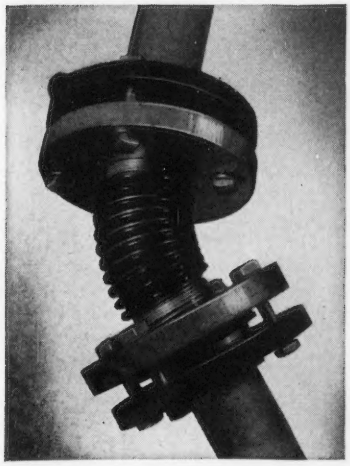

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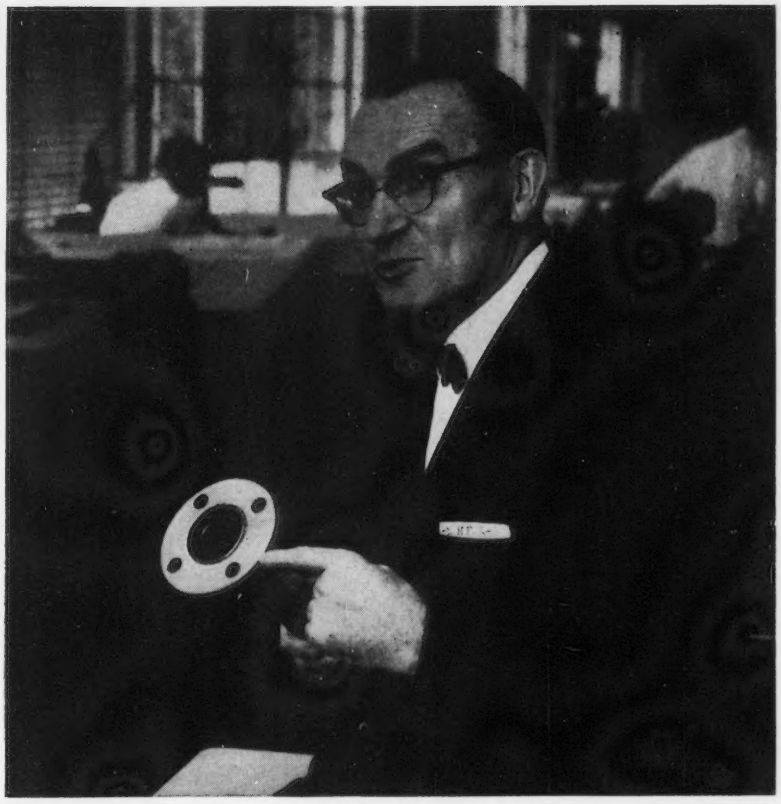

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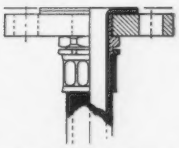

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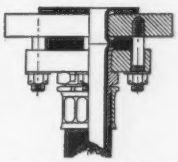

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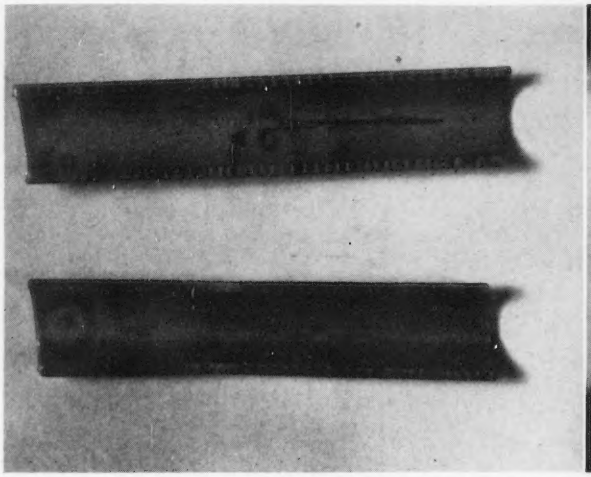

Figure 1—Pitting of Type 410 stainless condenser tube in recirculated cooling water.

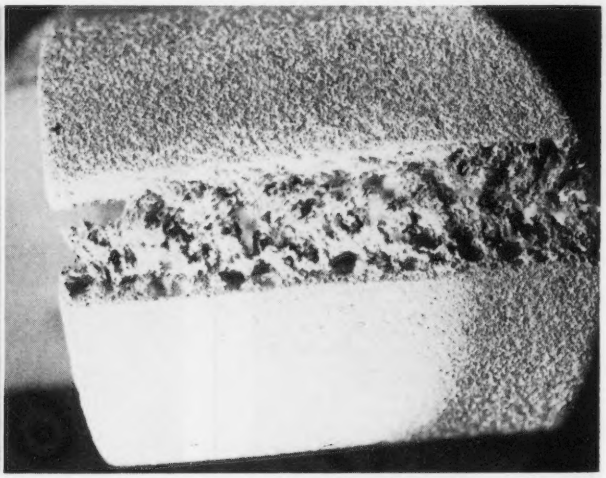

Figure 3—Selective corrosion of weld in 316 condenser tube in acetic acid service.

pose of these tests, much less their limitations.

These tests are based on the observed fact, which will be discussed in detail later, that the 18-8's are subject to intergranular corrosion when exposed in certain environments after certain heat treatments. Media employed in these tests are, from this standpoint, the most vicious known.

These quality control tests have *no established relationship to service performance.* For instance, fuming nitric acid has been known to cause intergranular attack on 304 of .06% carbon content which had passed both the Strauss and Huey Tests.[6] Also, while something is known of the effect of alloying elements on performance in boiling 65% nitric, there is no way of knowing how they will effect performance in other media except by exposure to them.

An outstanding characteristic of a quality control corrosion test is its ability to detect variations from specified chemical composition more exactly than will chemical analysis. For instance, the carbon analysis is accurate to ±.002% by the most refined techniques. Results from analyses by the ordinary laboratory technician may be accure to ±.005%.

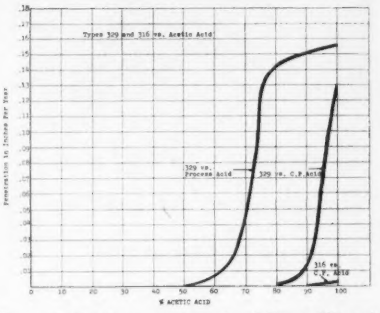

Figure 2—Relative corrosion rate of Types 329 and 316 vs acetic acid.

Further, carbon content in austenitic stainless steel may double from ladle-to-ingot-to-finished-sheet.

A laboratory corrosion test, however, will indicate accurately the performance of the finished steel under those conditions in which the carbon content and heat tratment are critical. Another important characteristic of such tests is their ability to detect metallurgical conditions which cannot be established by chemical analysis. For instance, columbium additions to austenitic stainless steels, as in Type 347, even when made in the proper proportions, may be inactivated by unusual amounts of nitrogen (for which an analysis is not normally made) or by improper heat treatment. In the former case, columbium is tied up in the form of the nitride, because it has greater affinity for nitrogen than for carbon. In the latter case, columbium carbides may be redissolved and redistributed leaving local areas unprotected. This is the mechanism of "knife-line attack," found in Type 347 stainless steel. In either case, the corrosion tests will indicate that columbium, although present in the specified amount, has not stabilized the alloy.

There is some overlap as to what can be established by corrosion tests as compared with metallographic examination. For instance, either will detect carburization, such as might occur acci-

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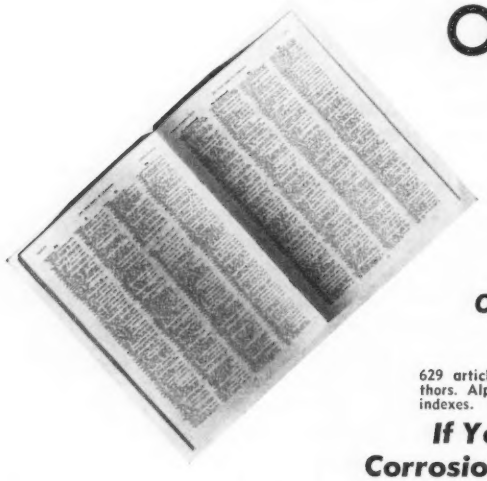

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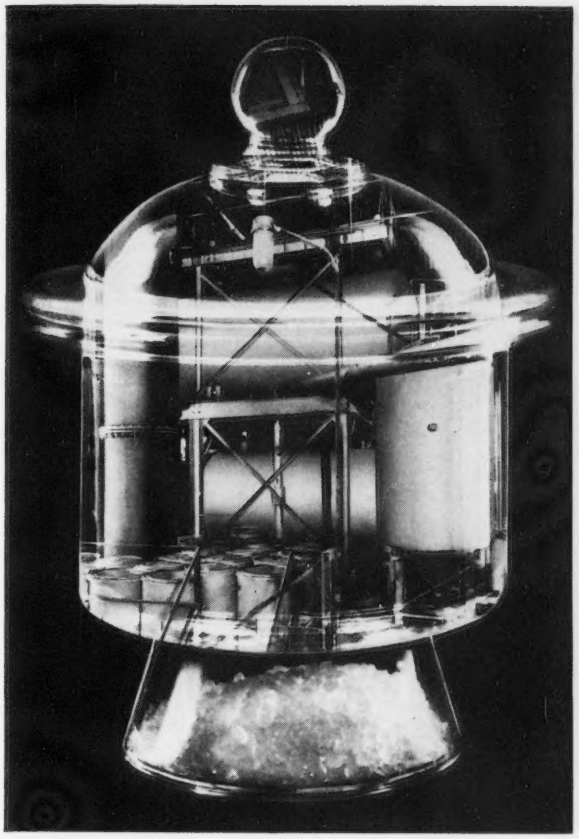

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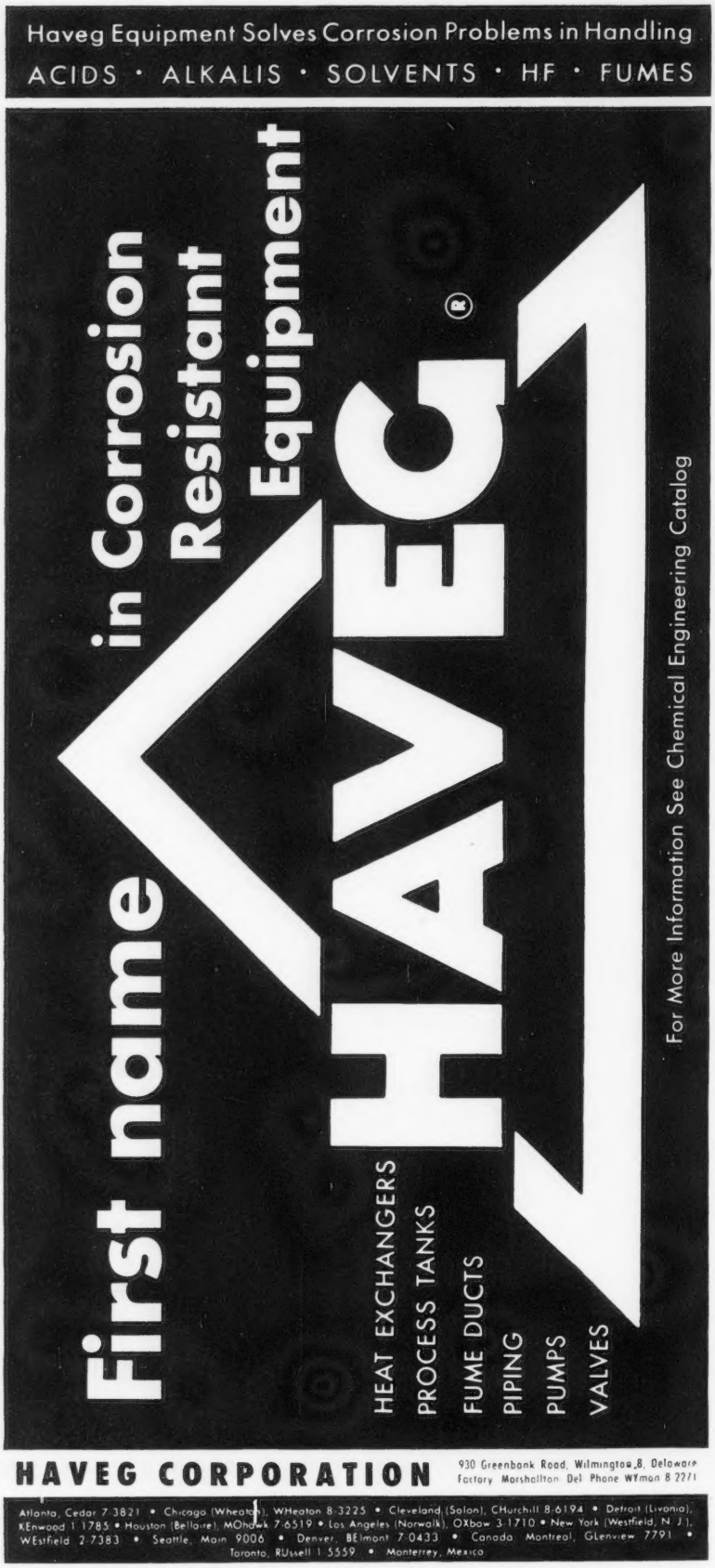
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dently during fabrication. Further, although the Oxalic Acid Screening Test is specified only for Types 304, 304-L and 316, in many cases it is possible that a metallographic examination after such a test might do all that the other quality control corrosion tests do, with less consumption of time and effort and at the same time be applicable to all grades of austenitic stainless steel. A considerable amount of work on the use of the Electrolytic Oxalic Acid Screening Test has been accomplished.[4]

It is of interest to discuss briefly the behavior of some alloys subjected to corrosion tests commonly called for in qualification tests or specifications.

It is frequently specified that 304 will pass the Huey Test in the as-received condition. Failure to do so indicates improper quench-annealing, which can be verified by re-testing after proper heat treatment. Type 304-L is required to pass the Huey Test after sensitization for one hour at 1250 F to precipitate carbides at the grain boundaries. Failure to do so indicates that the carbon has not been held to the required minimum value and the alloy might become subject to intergranular corrosion.

Type 347 is required to pass the Huey Test after sensitization, as for Type 304-L. Failure to do so may be due to too high an anneal, or to too high a nitrogen content and consequent ineffectiveness of the columbium addition. Obviously it also may be due to inadequate columbium content. Practically any such defect renders the material inacceptable provided it is known that the service requires the use of a stabilized grade. A fourth type of defect might be the presence of delta ferrite due to a slight imbalance of the usual austenitic structure. This ferrite would transform to sigma at the sensitizing temperature and also would be attacked by the nitric acid environment. However, the presence of ferrite would not necessarily mean that the material was unacceptable. Unless the annealed material showed slight traces of magnetism, it should be assumed that the poor performance in the Huey Test is due to some fault in the addition or activity of the stabilizing element.

The Huey Test is not applicable to the molybdenum-bearing stainless steels.[5] While it is possible that fully annealed Type 316 may pass the Huey Test, failure to do so does not mean it is off-quality material. The procedure is completely inapplicable to testing for sensitization of 316-L or 318 because the Huey Test is not capable of differentiating between intergranular corrosion due to carbide precipitation and intergranular corrosion due to sigma. There is, at present, no good qualification test for the molybdenum-bearing grades, with the possible exception of the Electrolytic Oxalic Acid Etch Test, although the Acidified Copper Sulfate (Strauss) Test is specified frequently. The writer feels that the Strauss Test will not detect intergranular susceptibility unless it is run for an unusually long time. The Strauss Test always should be followed by a 180 degree bend to check performance. Failure of 316-L or 318 to pass the Strauss Test is indicative of a higher carbon content than is permitted, or a deficiency or inactivity of columbium, or too severe a bend. This last

(Continued on Page 132)

ASPHALT * MOLTEN CAUSTIC * CHOCOLATE * COAL TAR PRODUCTS * HEAVY CRUDE OILS * FATTY ACIDS * GUMS * IMPREGNATING COMPOUNDS * MAELIC ANHYDRIDE * MOLASSES * HEAVY OILS * HYDROGENATED OILS * PARAFFIN * PETROLEUM RESIDUES

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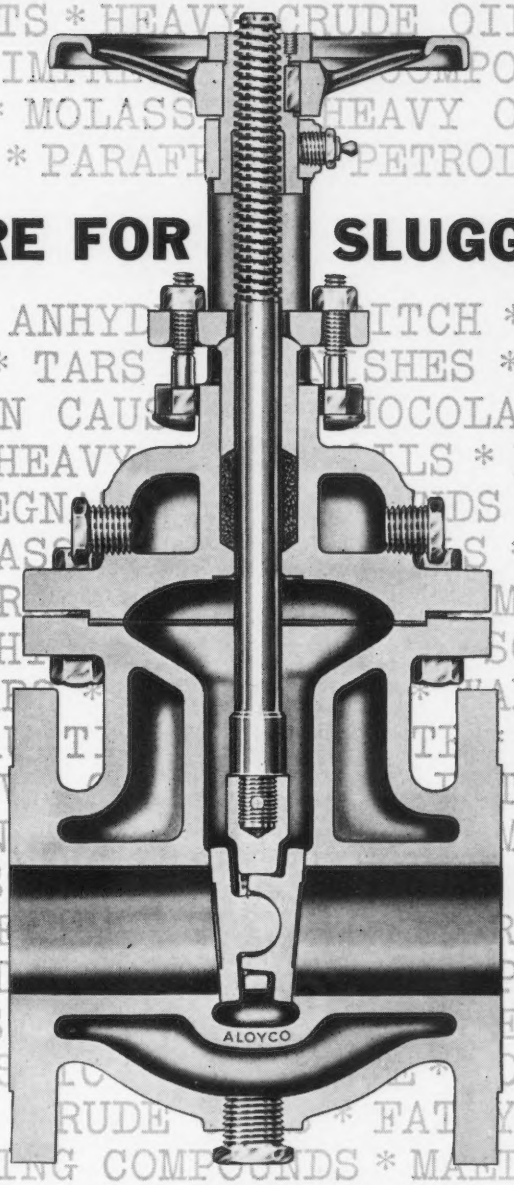

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possibility can be checked by a bend test on an annealed specimen.

Many specifications call for a series of quality control corrosion tests on welded specimens. The data so derived are difficult to interpret for the following reasons:

a. The heat-affected zone is small in relation to the rest of the specimen. Any difficulty, such as carbon pickup in welding a low carbon grade, would have to be a gross aberration in order to be adequately detected by weight loss.
b. The welding technique is not reproducible and may not be representative. This will be discussed in more detail later.
c. It is not possible always to determine whether the weight loss came from the parent metal, the heat-affected zone, or from the weld; unless checked by a supplementary metallographic examination.
d. Certain practices which are very beneficial for welding quality may lead to failure in these corrosion tests; for instance, the use of a slightly ferritic rod for the welding of 347.

It is believed that a separate evaluation of the quality of the plate and of the welding rod used would give more significant information. Low carbon electrodes should be checked by tests and the effectiveness of columbium additions to the stabilized electrodes should be verified likewise. However, welded specimens should not be used either for qualification tests or for other laboratory or field corrosion tests, because welded specimens do *not* faithfully reproduce conditions which obtain in welded process equipment.

Welded vs Seamless Tubing

While discussing specification of stainless alloys for process equipment, it might be well to consider an item which does not appear to be known generally. This is the use of welded versus seamless stainless condenser tubing. Specifications of the writer's company allow the use of welded stainless condenser tubing and nothing else has been used in its Texas City plant for the past six years. It is required, however, that the welded tubes be drawn after welding to a 35% reduction of area with a minimum of 15% reduction of wall. The tubes are annealed after drawing. This specification, which has raised a considerable number of questions among suppliers and fabricators, arises from the following considerations. It has been observed that in some process streams corrosion resistance of the cast weldment is inferior to that of the wrought parent metal of essentially the same composition. For this reason as-welded, undrawn stainless steel condenser tubes are not used. The 35% reduction in area and subsequent anneal works out the weld so that it is virtually indistinguishable from wrought material even under microscopic examination.

The writer believes a properly welded and drawn stainless condenser tube is equal in corrosion resistance to a seamless tube, so there is no reason to pay a premium for seamless stainless condenser tubing. This has been discussed with many manufacturers and none have been able to give any reason for preferring seamless instead of welded

(Continued on Page 134)

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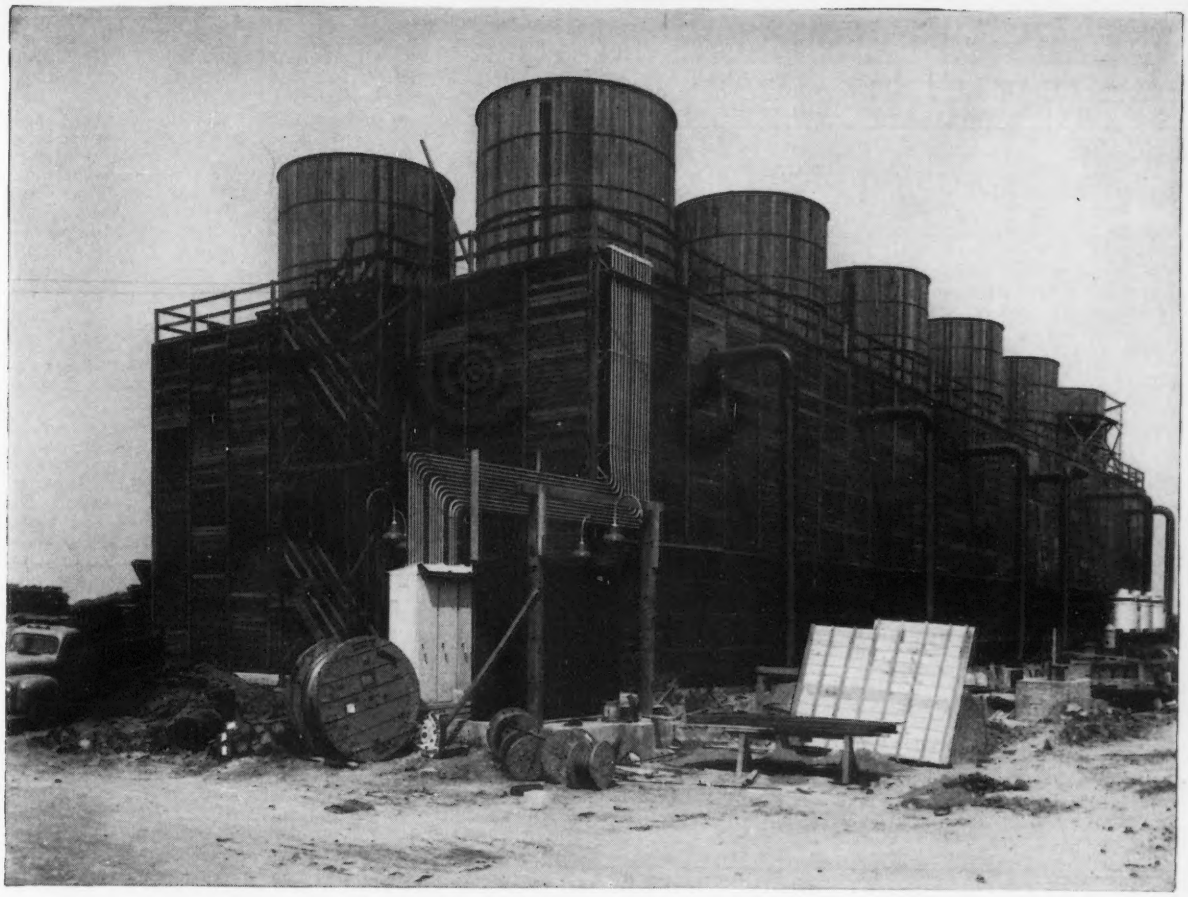

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TABLE 3—Low Temperature Impact Strength (Charpy Keyhole Notch) on Type 304 Welded With Type 308L Rod As Influenced by Various Heat Treatments

1750 F 2 HR. WATER QUENCH		1750 F 2 HR. AIR COOL		1250 F 2 HR. AIR COOL	
Weld	Heat-Affected Zone	Weld	Heat-Affected Zone	Weld	Heat-Affected Zone
51	.	37	88	17	48
47	63	34	65	15	45
49	84	32	90	18	43
46	65	52	98	20	76
44	63	55	92	20	63
59	72	50	85	18	84

and drawn tubing, except that some users prefer seamless despite the added cost. Recently, supercritical inspection of both welded and seamless tubing has shown defects which raise some question as to the applicability of either in critical services such as atomic energy installations. For normal chemical plant work, there seems to be no reason why good quality welded tubing should not be interchangeable with seamless condenser tubing. The writer's company has experienced stress cracking difficulties with seamless tubing when it was not fully annealed. This difficulty can be expected also with welded and drawn tubing unless prevented by proper specification.

B. **Applications.** High temperature behavior of stainless steels will not be covered in this article. It is common, however, to employ stainless steel for low temperature service (because of its excellent ductility down to —300 F) and for normal corrosive environments in chemical plants. It is important to realize, however, that the criterion of a wrought stainless steel in low temperature service should be at the level of performance of the weld in a welded vessel. Table 3 shows the relative post-heat-treatment impact strength at —80 C of the weld and parent metal in 304 plate welded with 308-L electrodes. It demonstrates the markedly lower ductility of the weld and a relatively minor effect of a stress relief. It is readily seen that the impact resistance of the welded area is lower than that of the parent metal. However, only deliberate sensitization at 1250 F diminishes the impact strength significantly and even then the material will pass ASME code requirements. Normal 18-8 castings are suitable for valves and pumps in low temperature service. In 18-8-3 castings (CF-8M), up to 10% free ferrite does not impair low temperature impact strength. To the best of the writer's knowledge and experience, there is no need in low temperature service for the extra low carbon or stabilized grades.

In corrosive environments, at the temperatures normally encountered in chemical processes, it is difficult to generalize on behavior even in a given medium. Lee[7] has an excellent description of a number of well established processes and the performance of various materials of construction. Some outstanding features of stainless steel applications are given below:

1. *Acetic Acid Environments*

An excellent review of acetic acid corrosion in general is provided by Teeple.[8] Other publications give data on performance of the stainless steels in such media.[2,4,7,9] Experience shows any conflicting data in the literature indicates process streams vary in their reactions with materials. Moreover, apparently identical streams may act quite differently because of the effect of minor or trace constituents which overshadow the action of the acetic acid itself.(1) Rarely, in chemical plants, does one encounter "acetic acid." Rather, acetic acid is found with numerous and variegated minor constituents.

The molybdenum-bearing grades offer the broadest range of usefulness in acetic acid environments, particularly if formic acid is also present.[8] However, 304, either annealed or sensitized, has been found equivalent to 316 in certain acetic acid process streams.

A peculiar difference between Types 329 and 316 is shown in Figure 2. The rate in boiling refined acetic acid rises quite sharply for 329 and much sooner than does the attack on 316. This apparently is not due entirely to the duplex (unbalanced) nature of 329, because unbalanced 18-8-3 cast tube sheets, valves and pumps are successfully used in many acetic acid applications. On the other hand, a wrought 329 corrosion coupon was observed to fail by stress corrosion cracking about the stamped identification marks after 15 months' exposure at 118 C in the make of an acetic acid still.

It is not unusual, however, to find that a cast structure may have less resistance than the corresponding wrought material. This has been mentioned previously in connection with comments about seamless versus welded condenser tubing. Figure 3 shows a weld in a 316 condenser tube, which had not been drawn after welding, corroded out entirely. On the other hand, hot acetic acid process streams are encountered in which the resistance of stainless is not significantly affected by heat treatment, duplex structure, molybdenum content or by cast, as compared with wrought, structure.

Special iron-chrome-nickel austenitic alloys of higher alloy composition than the 18-8's frequently are used for valves and pumps. These special alloys are exemplified by the 20 Type castings, by the wrought Carpenter 20 and by the Worthite and Durimet compositions. Uhlig[2] cites these special alloys as being resistant to all strengths of acetic acid, as well as to the vapors and to acetic anhydride up to the boiling point. Luce[10] cites these special alloys as having vastly superior resistance in hot acetic acid. The writer's experience indicates that while there are acetic acid process streams in which these highly alloyed austenitic alloys are markedly superior to the 18-8's, this is not invariably the case and actual corrosion resistance

(1) Temperature and degree of contamination must be taken into account.

(Continued on Page 136)

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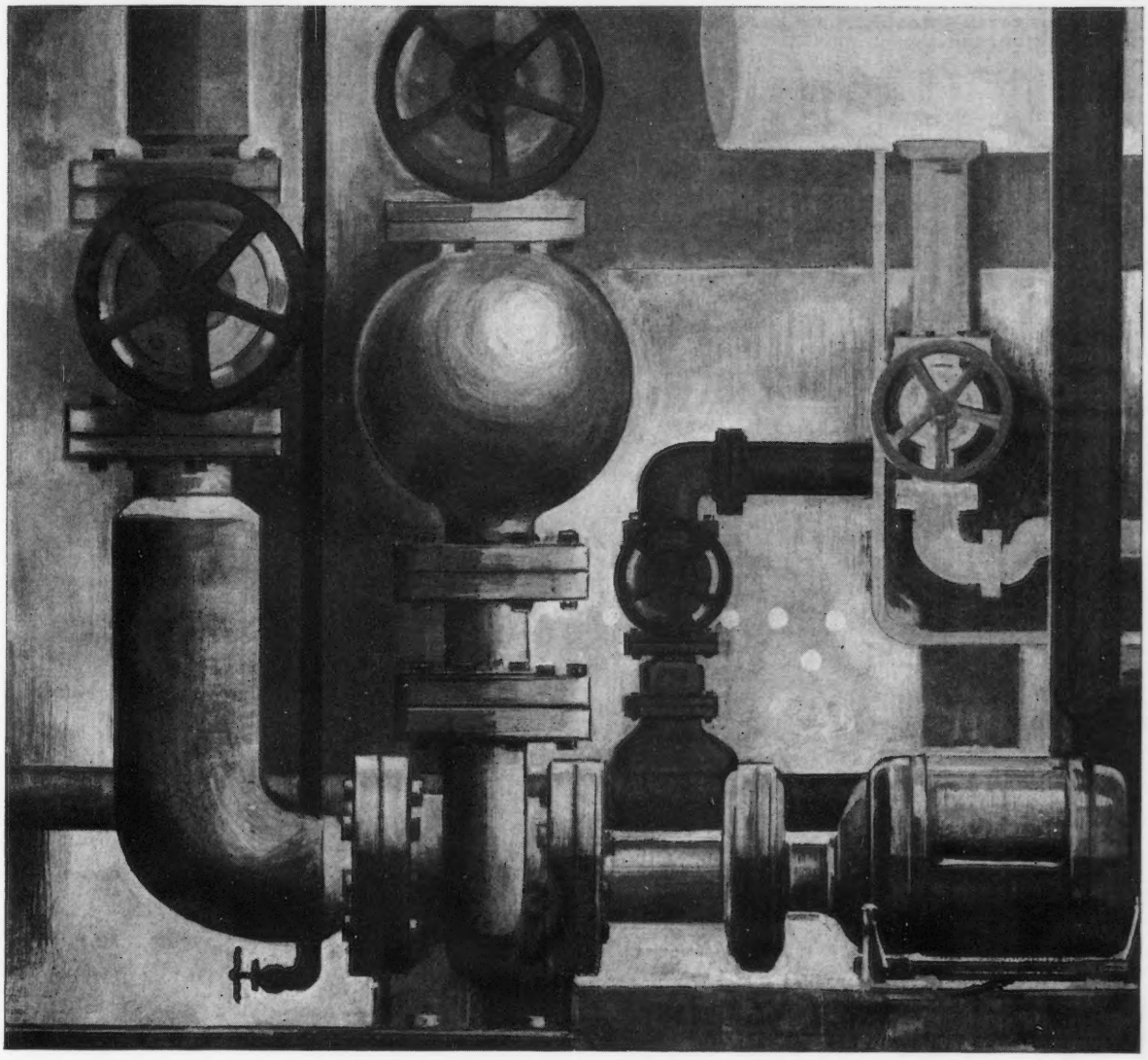

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should always be determined by appropriate field corrosion tests.

2. *Sulfuric Acid Environments*

Compared with their service in other process environments, stainless steels have limited application only in sulfuric acid media. An excellent discussion of many aspects of this service has been published by Kiefer.[11]

Applications of stainless steel in sulfuric acid solutions hinge on the determination of the passive-active boundaries for stainless steel. This, in turn, is a function primarily of the oxidizing capacity of the acid. On the other hand, an expression of the oxidizing capacity of sulfuric acid can not be divorced from a consideration of the materials of construction. For example Uhlig[2] mentions the change in corrosion resistance of copper in sulfuric acid at a concentration of 5 normal (25%). Kiefer,[11] on the other hand, determines the oxidizing capacity with respect to stainless to become measurable at 25 C and 8.9% acid. However, this also is a function of temperature and contaminants.

The use of even the molybdenum-bearing grades of stainless steel usually is limited to applications below 150 F and below 10% sulfuric acid. In acid strengths higher than 80% sulfuric acid, it is possible to use stainless steel again, although this is not commonly done because of the good resistance of ordinary steel and cast iron to the concentrated acid. In this application, for runs of pipe in which cast iron is unsafe and steel unsuitable, it is common to use the austenitic stainless steels, either 304 or 316 being satisfactory.

In intermediate strengths of sulfuric acid, from 30% to 80%, corrosion resistance of stainless steel is determined by inhibitors or oxidizing agents. It is common knowledge that relatively small amounts of cupric ions (on the order of 500 to 2000 ppm) can render moderate strengths of sulfuric acid noncorrosive to even the non-molybdenum-bearing grades of stainless steel. On the other hand, it would not be wise to rely on this in many services, because of the danger of accelerated attack at crevices where the metal would not have free access to the inhibitor. Another peculiar effect in relation to common concepts about oxidizing and reducing conditions, and their effect on the corrosion of stainless, is that Kiefer[11] has observed that stannous sulfate, which we would ordinarily think of as a reducing agent, has an inhibitory action on the corrosion of stainless steel in sulfuric acid almost equivalent to that of cupric sulfate (which normally is considered a relatively strong oxidizing agent).

It is in sulfuric acid media that the "super stainlesses" (or the specially alloyed iron-chrome-nickel-moly-copper stainless steels) come into their own. These special alloys were developed specifically for improved resistance in sulfuric acid media.[10] On the other hand, it should be evident that the pronounced effect of contaminants underlines the need for carefully designed laboratory and field corrosion tests before any stainless is used in sulfuric acid environments. Those environments in which sulfuric acid is only one of several major constituents should be viewed cautiously.

3. *Caustic Environments*

Austenitic stainless steels have good corrosion resistance in weak bases, such as ammonium hydroxide and certain organic amines.[4] Normally they show slight attack in solutions up to 50% caustic at temperatures up to about 220 F.[2, 4, 12, 13]

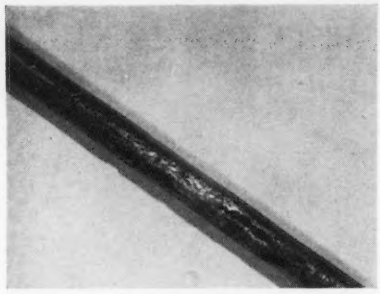


Figure 4—Localized-corrosion of Type 316 condenser tube in lean 20% monoethanolamine solution.

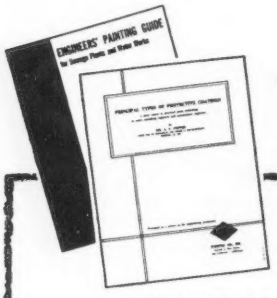
Behavior of austenitic stainless steels in caustic environments is governed primarily by the fact that strong alkalies at elevated temperatures tend to activate the metal. Under such circumstances it has been found that the 18-8's may have a lower resistance than ordinary steel.[13] It would be expected that certain galvanic effects or contaminants may bring about this adverse effect at lower temperatures and concentrations than are cited in the literature.(2) Occasionally, in actual services, instances of failure of both austenitic stainless steels and martensitic or ferritic grades have been encountered in strong caustic, which can be attributed only to this activation phenomenon.

In many amines services, molybdenum-bearing stainless steels usually are several hundredfold more resistant than their 18-8 analogues. It is not certain whether this resistance is due only to the presence of the molybdenum itself or to an indirect effect upon the borderline passivity of the alloys in strong organic alkali.

A practical and common problem which might well be included in this section of the discussion is the use of the stainless steels in ethanolamine acid gas scrubbing systems. These systems, which employ either an aqueous or a glycol solution of mono-, di- or triethanolamine for the removal of volatile acidic constituents from hydrocarbon streams, are commonly constructed of steel and in many instances give trouble-free service indefinitely. However, under conditions in which these units are hard pushed, or operated with extremely high reboiler temperatures, or with high mole ratios of the acidic constituents to the ethanolamine, the corrosion resistance of steel or alloy-steels often becomes inadequate. In some cases the substitution of either 304 or 316 tubes for the reboiler tubes or for the amine-versus-amine heat exchangers has proved a practical solution. On the other hand, under conditions in which there is mechanical disturbance, by erosion or turbulence, of the film on the metal surface, extremely rapid localized

(2) It should be pointed out in the caustic environments that normal commercial caustic contains a high percentage of sodium chloride and many cases of stress corrosion cracking of stainless steel have been reported in caustic environments.

(Continued on Page 137)



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corrosion of Type 316 has been encountered, as shown in Figure 4. Under these conditions, in which the passivity of the metal is destroyed, even the corrosion resistance of a Type 316 stainless apparently is less than that of ordinary steel, perhaps because there are galvanic effects between the disturbed anodic area and adjacent passive cathodic areas.

In summary, the stainless steels are apparently reliable in dilute alkaline environments of ammonia or sodium or potassium hydroxides. However, for stronger alkalies and in the organic ammonia derivatives, their resistance will have to be determined for each case. The possibility of activation and attendant vastly diminished corrosion resistance—even as compared with the ordinary steels—always should be considered.

4. *Sulfurous Acid Environments*

In sulfurous acid environments one would expect austenitic stainless steels to be resistant because of the oxidizing nature of the acid. In practice, however, the non-molybdenum-bearing grades frequently are pitted and the molybdenum-bearing grades are preferred.[4,7] At high temperatures, or in contaminated sulfuric acid it is necessary frequently to use the special super austenitic alloys, exemplified by Worthite and the 20-Type materials.

5. *Phosphoric Acid Environments*

In phosphoric acid environments molybdenum-bearing grades of 18-8 and the special high alloy austenitic stainless steels are often preferred materials of construction. Uhlig[2] cites 316 stainless steel as a Class A material up to 25% acid at the boiling point and to 85% acid at 95 C. At room temperature, Types 302 and 304 are satisfactory.[4] Lee[7] emphasizes that practical problems in the handling of phosphoric acid are complex, particularly because of the fluorine content of the phosphate rock from which the acid is derived and the sulfuric acid used in treating the rock.[3] In laboratory tests on digester liquors, in the range from 180 to 210 F and containing from 4 to 12% sulfuric acid, scattered data are obtained, apparently due to border-line conditions. However, overall results indicate that sulfuric acid concentration in digester liquor is of more importance than temperature, under the conditions tested. Corrosion in digesters can be minimized by maintaining low sulfuric acid concentrations, by assuring adequate mixing in the digesters and by controlling the temperature of the digester liquid.

McKay and Worthington[12] say that corrosion of austenitic stainless steels in phosphoric acid is "extremely sensitive to unknown factors." It is known that oxidizing ions inhibit, that inhibitors like arsenic may be added and that halides, or the intermediate oxides of phosphorus, may lead to accelerated attack. Sensitization probably is a factor. The writer has been told that molybdenum content is critical and that low molybdenum 316 (1.75 to 2.25% moly), advocated during the recent Korean crisis, corrodes rapidly under con-

(3) For storage of commercial grade, concentrated phosphoric acid, several manufacturers emphasize the need for high molybdenum (over 2.25%) and low carbon (under 0.06) preferably extra low carbon grades of stainless steel.

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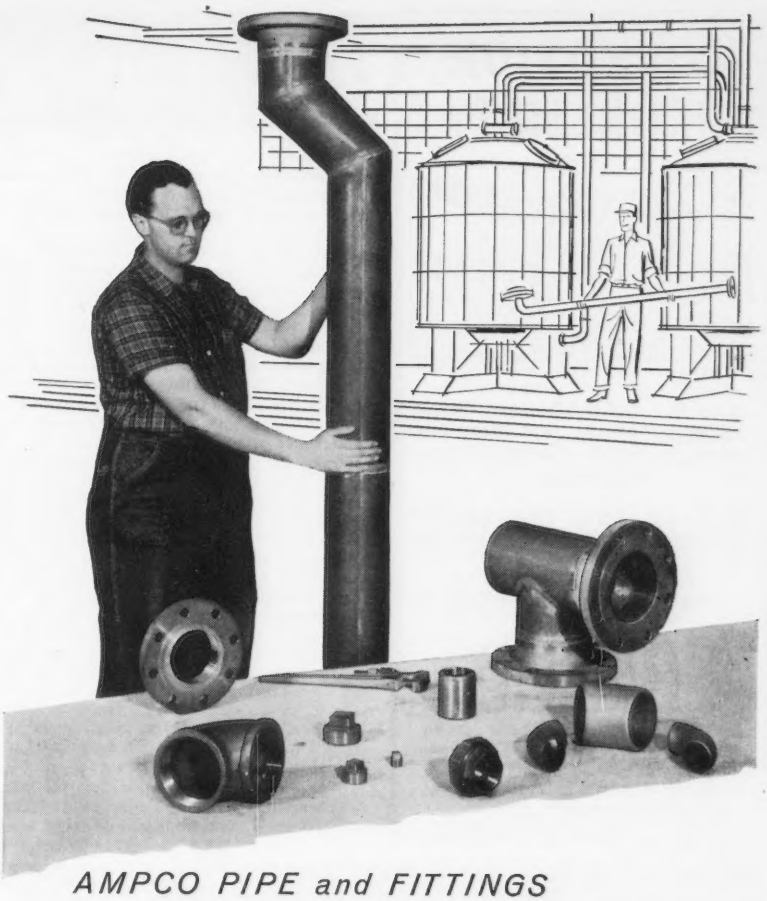

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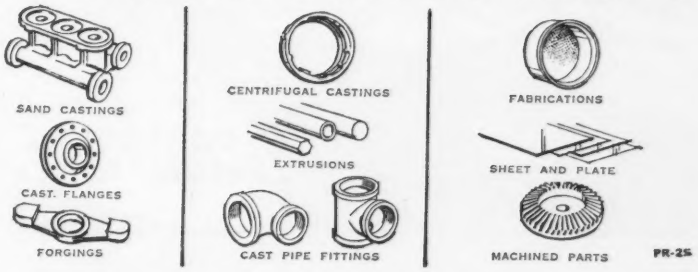

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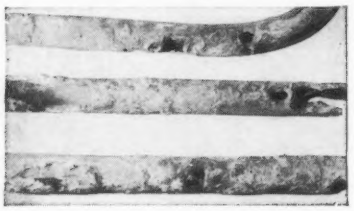
CORROSION PROBLEMS?



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6. Nitric Acid Services

Resistance of austenitic stainless steels to nitric acid at practically all concentrations and temperatures is widely known and hardly requires extensive comment. Stainless steel equipment is widely used in the production of dye-stuffs, explosives and in many processes involving nitric acid.

The adverse effect of heat treatment in the sensitizing range, or of welding, which effects similar changes, is widely known also. Early failures in nitric acid environments caused the development of the present qualification test in boiling 65% nitric acid. Columbium- and titanium-stabilized, or extra low carbon grades, are mandatory in most nitric acid services. Molybdenum does not seem to contribute materially to corrosion resistance of the alloys and actually may be detrimental.

A little known phenomenon with regard to vapor phase corrosion over strong nitric and mixed nitric-sulfuric acids has been described.[14] The investigation arose from the observed rapid corrosion of a Type 347 stainless steel-clad tank used in the storage of a mixed acid of the composition 85% nitric acid, 13.5% sulfuric acid, 1.5% water. A laboratory investigation of the mechanism of attack in this and similar acids showed severe vapor phase corrosion of austenitic stainless steel above 95% *total acidity*. The attack was not dependent on chloride or metal ion contamination and sensitization of 304 was found to be a secondary factor.

A common use of nitric acid on stainless steel equipment is the so-called "passivating" treatment employed on most stainless construction. This treatment consists of a wash for 30 to 60 minutes in 20 to 40% by volume of concentrated nitric acid at 55 to 70 C. Uhlig[2] emphasizes that the major purposes of this treatment are to clean the surface of the stainless steel of contamination by free iron, which might promote corrosive attack, and to equipotentialize the surface. In practice, any dilute acid probably would be equally effective, so there is no reason not to clean molybdenum-bearing grades with inhibited hydrochloric acid.(4) Laboratory demonstrations show that excessively high corrosion rates are not caused by this treatment and that it does not promote stress-cracking. However, caution must be exercised in the use of inhibited hydrochloric acid for equipment in high temperature service because residual chlorides in crevices or gasket surfaces have been found to cause pitting and cracking at 400 F.[15]

7. Cooling Water Environments

Austenitic stainless steels are employed widely for handling cooling waters such as have been found corrosive to steel or copper. In general, it may be said that molybdenum-bearing grades are somewhat better than straight 18-8's in waters containing significant amounts of chloride. It also may be observed that, for all practical purposes, sensitization phenomena are not significant and it is unnecessary to use low carbon or stabilized grades. Further, in the writer's experience, welded tubing is the equivalent of seamless tubing for this application.

Straight chromium grades of stainless steels can be used successfully in many waters. In others, however, as previously noted, they are rapidly pitted due to their susceptibility to oxygen concentration cell attack and their inability to repassivate in brackish water.

An outstanding characteristic of the austenitic stainless steels in many waters is their susceptibility to stress corrosion cracking. This will be discussed later. It is sufficient to note here that there is no known satisfactory solution to this problem inasmuch as the straight chromium grades tend to pit and little is known of the potentialities involved in the use of such things as the duplex alloys or the chromium-nickel-manganese alloys. However, most stainless steels are subject to cracking in some environments and even those grades upon which many have pinned hopes ,such as the duplex and manganese alloys, have been observed to crack in salt solutions or in salt spray tests. Screening tests are being initiated by NACE's T-5C-1 Corrosion in Gulf Coast Cooling Waters, on the susceptibility to stress corrosion cracking in brackish waters of the commonly used stainless alloys at various levels of stress.

C. **Use Against Contamination.** It should be apparent that where the stainless steels are employed merely to avoid contamination of foodstuffs or refined products, it is unnecessary to use low carbon or stabilized grades in seeking ultimate corrosion resistance. Substitute alloys 201 and 202 which have been proposed recently, as well as straight chromium grades, are of major importance if critical alloys are to be conserved.

(4) Note that ASTM A-380, "Recommended Practices for Descaling and Cleaning Stainless Steel Surfaces" for equipment fabricated from stainless is used extensively.

Part 2 will be published in October.

CORROSION ABSTRACTS

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CORROSION ABSTRACTS

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3. CHARACTERISTIC CORROSION PHENOMENA

3.5 Physical and Mechanical Effects

3.5.9, 6.2.4, 3.7.2

Oxidation of Chromium-Silicon Steels at High Temperatures. (In Russian.) G. M. Orlova and V. V. Ipat'ev. J. Applied Chem., USSR *(Zhur. Priklad. Khim.)*, **29**, No. 6, 819-828 (1956) June.

Effect of silicon on the kinetics of scale formation in the case of 6% chromium steel. Structure and composition of the scale on this steel alloyed with silicon.—BTR. 12840

3.5.9

The Craze-Cracking of Metals. L. Northcott and H. G. Baron. *J. Iron Steel Inst.*, **184**, Pt. 4, 385-408 (1956) Dec.

Craze-cracking is a form of surface failure produced by repeated thermal stresses. Cracking produced in 18-8, B.S. En 25 steel, 0.56 nickel steel and Armco iron at temperatures over 700 C is described. Graphs show stresses and strains set up in low alloy steels during various thermal cycles. Table shows room temperature thermal properties of the more common metals. Photomicrographs, 40 references.—INCO. 13339

3.5.9

Materials for Very High Temperatures. R. Steinitz. Schwarzkopf Development Corp. *Powder Met. Bull.*, **7**, 155-158 (1956) April.

Survey of metals available for use in high temperatures is made. Oxides are considered but are unstable, losing oxygen below the stoichiometric ratio. Carbides, borides and silicides can withstand sudden heating and cooling but they lack oxidation resistance. Other combinations and pure metals are considered.—NSA. 13396

3.5.9

New High Temperature Intermetallic Materials. Ronald Silverman. American Electro Metal Corp. U. S. Wright Air Development Center, Tech. Rept. 53-190 (Pt. 4), March, 1956, 48 pp.

The 100-hour stress-to-rupture life of alloy 223 (chromium-molybdenum-silicon 81/16/3) at 1800 F is in the range of 30,000 to 40,000 psi. Impact resistance is low, however, materials with appreciable amounts of sigma in the cobalt-chromium-molybdenum system appear to be unsuitable for jet engine turbine blading due mainly to their low stress-to-rupture life at 1800 F and low impact strength at room temperature. "Pearlitic" structures and preferred precipitation patterns were observed in many alloys of this type. The "pearlitic" lamallae are believed to be due to carbide formation, while the preferred precipitation pattern is believed to be due to the sigma phase. Of the alloys tested in the cobalt-zirconium, nickel-zirconium, vanadium-iron, vanadium-cobalt, vanadium-nickel, vanadium-silicon, manganese-cobalt, manganese-molybdenum and molybdenum-cobalt systems, alloys in the nickel-zirconium binary seem to have the most promise on the basis of oxidation resistance and ductility. X-ray patterns and photomicrographs were obtained for many of these alloys. (auth).—NSA. 13355

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AWWA—Journal, American Water Works Association, Amer. Water Works Assoc., 521 Fifth Ave., New York 17, N. Y.

BL—Current Technical Literature, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

BTR—Battelle Technical Review, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio.

CBEC—Centre Belge d'Etude de la Corrosion (CEBELCOR), 17 re des Drapiers, Brussels, Belgium.

CE—Chemical Engineering, McGraw Hill Publishing Co. 330 W. 42nd St., New York 18, N. Y.

EL—Electroplating. 83/85 Udney Park Road, Teddington, Middlesex, England.

EW—Electrical World, McGraw-Hill Publishing Co. 330 W. 42nd St., New York 18, N. Y.

GPC—General Petroleum Corp. of California. 2525 East 37th St., Los Angeles 11, Calif.

IIM—Transactions, The Indian Institute of Metals, 23-B, Notaji Subhas Road, P. O. Box 737, Calcutta, India.

INCO—The International Nickel Co., Inc. 67 Wall Street, New York 5, New York.

IP—Institute of Petroleum. 26 Portland Place, London W#1, England.

JSPS—Japan Society for the Promotion of Science, Address: Mr. Hayata Shigeno, Secretary, Committee of Preventing Corrosion, c/o Government Chemical Industrial Research Institute, 1 Chome Nakameguro, Meguroku, Tokyo, Japan.

MA—Metallurgical Abstracts, Institute of Metals, London, England. 4 Grosvenor Gardens, London SW 1, England.

MI—Metallurgia Italiana, Associazone Italiana di Metallurgia. Via S. Paola, 10, Milano, Italia.

MR—Metals Review, American Society of Metals. 7301 Euclid Ave., Cleveland 3, Ohio.

NALCO—National Aluminate Corp. 6216 West 66th Place, Chicago 38, Illinois.

NBS—National Bureau of Standards. Supt. of Documents, U. S. Gov't Printing Office, Washington 25, D. C.

NSA—Nuclear Science Abstracts. United States Atomic Energy Commission, Technical Information Division, Oak Ridge, Tenn.

PDA—Prevention Deterioration Abstracts. National Research Council, 2101 Constitution Ave., Washington 25, D. C.

RAD—Reynolds Aluminum Digest, 2500 South Third Street, Louisville, Kentucky

RM—Revue de Metallurgie, Paris, France. 5 Cite Pigalle, Paris (9e), France.

RPI—Review of Current Literature Relating to the Paint, Colour, Varnish and Allied Industries, Research Association of British Paint, Colour & Varnish Manufacturers, London. Waldegrave Rd., Teddington, Middlesex.

SE—Stahl Und Eisen, Verlag Stahleisen, M. B. H., Dusseldorf, August-Thysen Str. 1. Postscheck Koln 4110, (22a) Dusseldorf, Germany.

TIME—Transactions of Institute of Marine Engineers. 85 The Minories, London EC 3, England.

UOP—Universal Oil Products. 310 South Michigan Ave., Chicago, Illinois.

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3.5.9

Effect of Gaseous Media at High Temperatures on the Corrosion of Alloys. (In Russian.) S. G. VEDENKIN AND E. L. KAZIMIROVSKAYA. *Metallovedenie i Obrabotka Metallov,* No. 5, 55-63 (1956).

A review of non-Russian work (1951-54) on the corrosion of materials (alloy steels, nickel-chromium, Hastelloys, alloy cast iron, etc.) at elevated temperatures. 14 references.—MA. 13430

3.5.9, 3.5.8, 2.1.1

Metals at Elevated Temperatures. R. W. GUARD. *Product Eng.,* **27,** No. 10, 160-164 (1956) Oct.

Diagrams of homologous temperature scale for nine common metals. Comparison of hot and cold behavior of metals using homologous scale. Comparison of creep and rupture tests under different conditions using Larson-Miller parameter permits the prediction of stresses in long-time service from short-time tests at higher temperatures. Relation between fatigue and stress rupture-failure; temperature dependence of thermal expansion coefficient and elastic modulus.—BNF. 13223

3.5.9, 6.3.11, 3.8.4

Oxidation and Volatilisation of Platinum at Elevated Temperatures. R. LACROIX. *Rev. Met.,* **53,** 809-818 (1956) Nov.

Microscopic study of surface phenomena during high-temperature oxidation of platinum. Formation of oxide crystals, intergranular grooves and striations were observed. Attempted theoretical explanation of these effects is given.—INCO. 13168

3.6 Electrochemical Effects

3.6.6

Galvanic Couple Corrosion Studies by Means of the Threaded Bolt and Wire Test. K. G. COMPTON AND A. MENDIZZA. Paper from "Symposium on Atmospheric Corrosion of Non-Ferrous Metals," ASTM Special Publication No. 175, 1956, 116-123; disc., 124-125. American Society for Testing Materials, 1916 Race Street, Philadelphia, Pennsylvania.

Concludes that no very broad generalization can be made as to the relative severity of dissimilar metal galvanic-couple corrosion; corrosion rate seems to be an arbitrary value.—MR. 12734

3.6.8

Fundamental Studies on Corrosion of Alloys. Rept. VIII. A Consideration on the Hydrogen Overvoltage of Alloys. N. OHTANI. *J. Japan Inst. Metals,* **20,** No. 8, 468-472 (1956) Aug.

Applying the results of statistico-thermodynamical theory on hydrogen absorption to hydrogen electrode reaction, the author expressed the heat of activation ΔH^* of the alloys which do not form any hydride by the following formula: $\Delta H^* = f(\rho, T) - \alpha Z' RT \ln \lambda_H{}^J J_H (N_A/N + N_{B/K}N) + \alpha(\psi_{AH} + \chi_A/2)$ where $\lambda_H{}^J$: the absolute activity of hydrogen atom in gas phase, J_H: the partition function of hydrogen atom, ψ_{AH}: the interaction energy between A atom in alloy AB and hydrogen atom, K: a term concerned with ψ_{AH} and ψ_{BH}. The variation of ΔH^* in iron and copper alloys and the validity of the results of the previous reports were discussed from the formula above mentioned.—JSPS. 12879

3.6.8

Determination of Parameters in Overpotential Studies. H. B. MORLEY AND F. E. W. WETMORE. *Can. J. Chem.,* **34,** 359-363 (1956) March. 13260

3.6.8, 3.2.2, 3.5.6, 5.8.3

Is There a Quantitative Relationship Between Hydrogen Overvoltage and the Solubility of This Gas in Metals? (In English.) M. SMIALOWSKI. *Bull. Acad. Polon. Sci.,* Classe iii, **4,** No. 1, 43-47 (1956)

Smialowski concludes that, in a polarized cathode of a metal M capable of dissolving hydrogen, the hydrogen first penetrates into M as H^+ and forms a compound MH_x which, after a certain stage of saturation has been reached, dissociates, giving atomic hydrogen, which then forms molecular hydrogen on the crystal surfaces; if the hydrogen cannot escape, the pressure rises until either M cracks or diffusion of atomic hydrogen begins. If M does not dissolve hydrogen, the rate-determining step of direct discharge of OH^+_3 on the cathode surface is followed by desorption and overvoltage (η) is high. When M dissolves hydrogen there is also the other mechanism, and η is lower, being controlled by the rates of both penetration of H^+ into M and desorption; arsenic on the surface of iron retards the formation of molecular hydrogen from atomic hydrogen and raises η. Polar organic compounds adsorbed on M reduce the rates of both discharge of OH^+_3 and penetration of H^+, and therefore increase η. 15 references.—MA. 13349

3.7 Metallurgical Effects

3.7.3, 6.2.3

Isothermal Studies on the Weld-Metal Microcracking of Arc Welds in Mild Steel. A. E. FLANIGAN AND Z. P. SAPERSTEIN. Paper before Am. Welding Soc., Nat'l. Fall Mtg., Chicago, Nov. 1-5, 1954. *Welding J.,* **35,** No. 11, 541s-556s (1956) Nov.

Attempt was made to determine temperature-dependence of time required for isothermal immunization against weld-metal cracking for E6010 welds on A285-C firebox-quality mild steel. Immediately after welding, specimens were subjected to isothermal treatments at 1700-212 F and then quenched in room temperature water to evaluate their cracking resistance. Time required for immunization has maximum value for specimens treated at 875 F. At 750-212 F immunization times were short and nearly independent of temperature. Only few cracks were found in welds quenched right after welding without intervening isothermal treatment. No correlation existed between weld-metal hardness and incidence of microcracking. Susceptibility was absent initially but tended to develop in temperature range somewhat below A_1 if cooling rate was slow. Hydrogen-retained austenite hypothesis concerning mechanism of weld-metal microcracking is discussed in light of the experimental data. Graphs, photomicrographs, 27 references.—INCO. 13205

3.7.3, 6.2.4

Upper Nose Temper Embrittlement of a Nickel-Chromium Steel. L. D. JAFFE AND D. C. BUFFUM. Paper before AIME, Cleveland, October, 1956. *J. Metals* (Trans. AIME), **9,** No. 1, 8-16 (1957) Jan.

Temper embrittlement of SAE 3140

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was investigated isothermally and with temperature changes. Various heat treatments used are tabulated. Embrittlement was most rapid at 490-550 C range and just below the Ae_1 near 675 C. Embrittlement in lower range showed rapid grain boundary attack by etheral picric acid and fracture along austenite grain boundaries: at 675 C attack by picric acid was slow and fracture occurred along ferrite grain boundaries. Embrittlement during slow cooling from 675 C appeared to be associated with lower range. Tables, graphs, photomicrographs. —INCO. 13185

3.7.3, 6.4.2

Jointing of Aluminium with Other Metals: Methods of Assembly. (In French and German.) E. ZURBRUGG. *Aluminium Suisse,* **6,** No. 5, 174-180 (1956) Sept.

Methods of assembly, corrosion and mechanical strength for joining the two metals (e.g., aluminum to copper, steels, etc.) by grooving, riveting (choice of rivets), bolting (insulating techniques), shrinking or glueing. Details of brazing and welding processes.—BNF. 13446

3.7.3, 7.2

What Causes Underbead Cracks in Welds? A. M. HILL AND F. W. ZILM. Paper before Am. Soc. Mech. Engrs., Petroleum-Mechanical Conf., Dallas, 1956. *Oil Gas J.,* **54,** No. 85, 108-112 (1956) Dec. 17.

Description of development of new maintenance-welding procedure for use on pipelines where pipe surface temperature is below 50 F. Hydrostatic tests proved that fittings which completely encircle pipe wall will withstand higher internal pressures than reinforced saddle type of fittings. New procedure outlines methods and materials to be used on pipe. Tests showed that common method of repairing corrosion pits of common grades of pipe should be restricted to low and medium carbon steel grades. High yield-strength pipe was very sensitive to quenching effect of liquid contained in the chambers. Underbead cracking occurred in all except one test specimen made with E6010 and E7010 electrodes. Photos.—INCO. 13229

3.7.4, 3.8 2

On the Ordering Effects in the Corrosion of Cu_3Au by Aqueous Ferric Chloride. H. PAPAZIAN AND ROBERT A. LAD. *J. Metals* (Trans. AIME), **8,** Sec. 2, 1340 (1956) October.

Some recent results obtained in the study of the rates of corrosion of ordered and disordered polycrystalline Cu_3Au.—MR. 13338

3.7.4, 3.8.4

On the Influence of Crystal Orientation Upon Oxidation Rate. K. M. CARLSEN. *Acta Metallurgica,* **5,** No. 1, 58-60 (1957) Jan.

Uses explanation put forth for definnite geometric growth of oxide nuclei in oxide film to partly explain effect of orientation upon oxidation velocity. Conversion into oxide at interface metal-oxide occurs directionally by more or less consecutive jumps in certain energetically preferred lattice directions, resulting in difference in rate of jumps at metal-oxide interface of grains of different orientation. Views outlined envisage concentration difference which is constant with respect to time but differs from grain to grain; this disagrees with Wagner's formula for oxidation rate based on constant concentration difference across oxide film.—INCO. 13197

3.7.4, 4.3.2, 6.2.3

Effect of Steel Structure on Its Solubility Rate in Acid. (In German.) OTTO NIEZOLDI. *Chem. Ztg.,* **80,** No. 20, 721-723 (1956) October 20.

Concludes that corrosion behavior in hydrochloric acid is influenced by the primary crystal structure.—BTR. 13340

3.7.4, 3.8.4, 6.3.6

The Structure of Oxide Films on Different Faces of a Single Crystal of Copper. (In English.) KENNETH R. LAWLESS AND ALLAN T. GWATHMEY. *Acta Metallurgica,* **4,** No. 2, 153-163 (1956) March.

The effect of many variables on the structure and orientation of oxide films on eight crystal faces of copper was investigated. Cuprous oxide formed initially on all faces, but cupric oxide was formed above a certain thickness. The type of orientation varied with crystal face and the degree of orientation with pressure, temperature and oxide thickness—being greater for higher temperature and lower pressure. The factors controlling the orientations were: 1) At least one pair of (110) directions in copper and cuprous oxide must be ||. 2) For low-index planes a good fit between close-packed rows is more important than parallelism of close-packed planes. 3) In the case of high-index planes the orientation is determined by the major faces near the high-index face [the (111) are preferred to (110)]. Cupric oxide was not oriented when present. 16 references.—MA. 12869

3.7.2, 6.4.2

Influence of Titanium and Boron on the Chemical Resistance of Refined Aluminum (99.9%). (In French.) M. GYENES-HOLLO. *Corrosion et Anticorrosion,* **4,** No. 2, 47-52 (1956) Feb.

Results of investigation on the influence of small additions of titanium (0.01-0.6%), boron (0.01-0.9%) and titanium plus boron (0.02-0.165% titanium, 0.04-0.20% boron) on corrosion of commercially-pure aluminum in dilute sulfuric acid, nitric acid, hydrochloric acid and sodium hydroxide: figures for pure (99.99%) aluminum are included as comparison. Results are discussed.—BNF. 12004

3.8 Miscellaneous Principles

3.8.2, 1.6

Electrochemistry: Principles and Applications. E. C. POTTER. Book, January 15, 1957, 418 pp. The Macmillan Company, 60 Fifth Ave., New York 11, New York; Cleaver-Hume Press Ltd., 31 Wright's Lane, Kensington, London W.8.

A text intended to acquaint the technical student and the specialist electrochemist with some of the theoretical and practical aspects of electrochemical reactions. The author considers the laws of electrolysis, electrolytic conductance and transport and ionic activity; ionic equilibria, reversible and irreversible processes; reference electrodes and cells; electric double layer and electrokinetic phenomena, electrochemical measurements, theoretical and practical aspects of metallic corrosion; general principles of technical electrolytic processes, cathodic and anodic processes and electrochemistry as a source of energy in primary and secondary and fuel cells.

There is an alphabetical subject index and bibliography. 13194

3.8.4, 3.4.9, 6.3.21

Metal-Water Reactions. Part III. Kinetics of the Reaction between Thorium and Water Vapor. BRUCE E. DEAL AND HARRY J. SVEC. *J. Electrochem. Soc.,* **103,** No. 8, 421-425 (1956) August.

An examination of the reaction between thorium metal and water vapor has been made between 200 and 600 C and at water-vapor pressures of 25 to 100 mm Hg. Analyses of the products indicate that thorium dioxide and hydrogen are the main species formed during the reaction with formation of ThH_2 as a possible side reaction product. Two methods, one gravimetric and the other manometric, were used to determine rates of reaction. During these rate studies, the variables time, temperature and pressure were examined. Reaction data were found to obey the logarithmic rate law $w = k\log(1 + 0.45t)$. The activation energy was found to have an average value of 6.44 ± 0.75 kcal/mole. From marker experiments it is postulated that inward diffusion of water molecules or a related species is the rate-determining step of the reaction. Other experiments showed that hydrogen diffused into the metal in varying amounts depending on temperature and water-vapor pressure. This hydrogen was evolved when the samples were heated under high vacuum to 1200 C. 12723

3.8.4, 3.7.2

Oxidation of Alloys Involving Noble Metals. CARL WAGNER. *J. Electrochem. Soc.,* **103,** No. 10, 571-580 (1956) Oct.

Diffusion processes during the oxidation of alloys involving noble metals are analyzed theoretically. An oxide film of uniform thickness is stable only if diffusion in the alloy is relatively rapid compared with diffusion in the oxide of the less noble metal. Otherwise, there is the tendency to form a rugged alloy-oxide interface. The oxidation rate is calculated for the limiting case of a scale consisting of protruding sections of the oxide of the less noble metal interspersed with slender trunks of alloy rich in the more noble metal. Under these conditions, the oxidation rate of alloys containing up to 50% of noble metal is supposed to be of the same order of magnitude as the oxidation rate of the less noble metal in its pure state. 12633

3.8.4, 6.3.3, 6.3.10, 6.2.5

Photoelectric Study of Oxidized Chromium, Oxidized Nickel and Oxidized Chromium-Nickel Steel in Alkaline Solution. (In Russian.) I. B. RABINOVICH AND V. I. VESSELOVSKII. *J. Phys. Chem., USSR* (Zhur. Fiz. Khim.), **30,** No. 2, 313-320 (1956).—BL. 13324

3.8.4, 6.3.6, 3.2.3

Growth of External Copper Layers During the Internal Oxidation of Dilute Copper-Aluminum Alloys in a Cuprous Oxide-Copper Pack. D. L. WOOD. *J. Metals* (Trans. AIME), **8,** No. 10, Sec. 2, 1252-1256 (1956) October.

Experimental data suggest that the cuprous oxide is vaporized as molecules, which are dissociated at the oxygen-absorbing surface.—MR. 13456

3.8.4, 6.3.10, 3.7.4

Oxidation of a Monocrystal of Nickel. K. R. LAWLESS, F. W. YOUNG AND A. T. GWATHMEY. *J. Chim. Phys.*, **53**, 667-674 (1956) July-August.

Study of oxidation rates of different faces of nickel crystal, formation of nickel oxide nuclei and orientation relations between nickel and oxide.—INCO. 13156

3.8.4, 6.3.21

Formation of Antimony Suboxide (SbO) During the Burning of Antimony Metal. HELMUT HENNIG. *Z. Erzbergbau u. Metallhuttenw.*, **9**, No. 2, 56-57 (1956).

When metallic antimony is heated to 1000-1200 C and the burning metal poured on to a clean, white porcelain tile, many of the prills formed show three zones—a black core of metal, surrounded by a brown-grey zone of antimony oxide and a white shell, which, it is suggested, is antimony suboxide.—MA. 13240

4. CORROSIVE ENVIRONMENTS

4.4 Chemicals, Organic

4.4.2, 8.8.1

The Manufacture of Phthalic Anhydride by the Fluidized-Catalyst Method. H. L. RILEY. Paper before Soc. Chem. Ind., November 13, 1956. *Chemistry & Industry*, No. 49, 1464-1468 (1956) December 15.

Description of solution of two problems in design and development of fluidized-catalyst method of manufacturing phthalic anhydride. Fluidized reactors which use an expensive catalyst cannot tolerate any loss and it is therefore necessary to filter exit gases. This is particularly so with vanadium pentoxide catalysts, loss of which from a reactor would give rise to a health hazard. Porous ceramic filters were used in large fluidized-catalyst phthalic anhydride plants and porous stainless steel filters in pilot plants. As temperature falls below dew point of moisture in gas stream, condensate becomes highly corrosive owing to formation of free maleic and phthalic acids which necessitates use of stainless steel as constructional material. Description of new condensing method developed is given. Photos, diagrams, graph.—INCO. 13375

4.4.6, 4.2.3, 2.3.4

Fuels for High Performance Engines. J. G. WITHERS AND H. J. EATWELL. *Automobile Engr.*, **46**, No. 12, 521-525 (1956) Dec.

Discussion of properties of 100 octane number gasoline as to additives, antiknock quality, volatility and stability. Two tests used by British Petroleum Co. to measure tendency to form fuel deposits are described. With an unstable gasoline, products of partial combustion contain high proportions of gum and lacquer, some of which are deposited on piston assembly during passage of products of partial combustion into sump. Products that reach sump are then circulated with lubricating oil and tend to be deposited. New 100 octane number motor fuels have a balanced quality so that their volatility and stability are better than gasolines already available. Graphs, photos.—INCO. 13453

4.4.7, 4.4.6

High Temperature Lubricant Studies. E. ERWIN KLAUS AND MERRELL R. FENSKE.

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181a

Pennsylvania State Univ., Petroleum Refining Lab. U. S. Wright Air Development Center, Tech. Rept. 56-224, May, 1956, 33 pp.

Viscosity properties, lubricity, thermal stability, oxidation and corrosion of silicones, silicates, hydrocarbons, diesters and halogenated aromatic hydrocarbons from 500 to 700 F were evaluated in a high-temperature lubricant study.—NSA. 13436

4.4.7, 5.8.4

Hydroxyarylstearic Acids as Oxidation and Rust Inhibitors in Lubricants. H. GISSER, J. MESSINA AND J. SNEAD. Pitman-Dunn Labs. *Ind. & Eng. Chem.*, **48,** No. 11, 2001-2004 (1956) Nov.

Hydroxyarylstearic acids were prepared by condensation of phenols with oleic acids, using aluminum chloride. Compounds were active as oxidation and rust inhibitors in instrument oil compositions. Most effective compound prepared was hydroxyphenylstearic acid, which in 2% solution was an effective antioxidant (at 100 C) and rust inhibitor in bis (2-ethylhexyl) sebacate and triiso-octyl phosphate. Graphs, tables.—INCO. 13220

4.4.8, 8.4.3

Corrosion of Chemical Apparatus by Aqueous Solution of Mono-Ethanolamine. (In Russian.) M. L. RUTKOVSKII. *Khim. Prom.* (Chemical Industry), No. 3, 25-28 (1956) April-May.

Corrosion of carbon steel is initiated by corrosive compounds in the monoethanolamine. By distilling under vacuum the compounds can be eliminated.—MR. 13368

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4.5 Soil

4.5.1

The Kinetics of Cathodic Processes Involved in the Corrosion of Metals in the Soils. (In Russian.) N. D. TOMASHOV AND IU. N. MIKHAILOVSKII. *Doklady Akad. Nauk SSSR,* **107,** No. 6, 853-856 (1956) June.

Study of migration of oxygen in soil yields quantitative data for determining rate of cathodic processes responsible for corrosion of metals in various soils. Graphs. 8 references.—BTR. 12622

4.5.1, 3.3.4, 5.4.5, 3.6.9, 1.7.1

Corrosion of Cast Iron and Steel in the Soil. (In Dutch.) Publication No. 48, November, 1956, 26 pp. Metallinstituut TNO, Afdeling Corrosie, Delft, Holland.

A report on 25 years of study by Corrosion Commission II marking the 25th anniversary of the commission on October 15, 1956. Contents include: 25 Years of Corrosion Commission II; Discovery of Anaerobic Metal Corrosion in the Soil; Asphalt Bitumens; Practical Results of the Work Done by the Corrosion Commission; Comparisons of Stray Current in Soil; and Coal Tar. 13276

4.5.1, 3.6.6

Mechanism of Electrochemical Corrosion of Metals in Soil. (In Russian.) N. D. TOMASHOV AND IN. N. MIKHAILOVSKII. *Doklady Akad. Nauk SSSR,* **108,** No. 4, 668-671 (1956) June 1.

Effects of formation of corrosion couples on corrosive action.—MR. 13408

4.5.2, 2.2.6, 7.7

The Protection of Cable Sheathing: The Mechanism of Corrosion of Metal Pipes in Soils and Practical Methods of Prevention. W. W. ROBSON AND A. R. TAYLOR. *Chemistry and Industry,* No. 41, 1111-1119 (1956) October 20.

Specimens were buried in trenches representing clay, chalk, peat, made-up ground, sandy loam, brackish silt and ashes and were checked for corrosion in 1, 3 and 10 years.—MR. 13376

4.6 Water and Steam

4.6.9, 2.1.1

Calculation of the pH of Oil Well Waters. WALTER F. ROGERS. *Corrosion,* **12,** No. 12, 595t-601t (1956) Dec.

Studies of the causes of corrosion of oil well tubular goods by the produced fluids require a knowledge of the pH of the well water at the point in the well where corrosion occurs. The well head pH of such water is not necessarily the pH of the water within the well because of the loss of acid gases, such as carbon dioxide, through pressure reduction on the fluid during the course of production. It is necessary, therefore, to recombine into the brine the amount of volatile acidic material to determine the pH at the point in question.

This study has developed a technique for calculating the down hole pH through such recombination. The technique covers three types of well waters: 1) Oil well brines, the pH of which is controlled by the carbonic acid-bicarbonate ratio, 2) Oil well brines containing top acidity as the result of hydrolysis of calcium and magnesium salts or organic acids, and 3) Condensate waters. 12643

4.6.10, 5.4.8
Brine Fighter. E. J. RUBLE. *Paint, Oil, Chem. Rev.*, **119,** No. 9, 5 (1956).
Synthetic resin paints are far superior to red lead paints on surfaces subjected to brine drippings. Paints including vinyl resins, phenolic resins, chlorinated rubber, neoprene and epoxy resins have come through recent field tests with flying colors.—RPI. 13393

4.6.12, 8.4.3
Battle Against Ocean-Water Corrosion. C. H. JONES, JR. Long Beach Oil Development Co. *Oil Gas J.*, **54,** No. 82, 76-79 (1956) November 26.
Three years of experience with a trial flood in harbor area of Wilmington field showed that corrosion is a major problem when sea water is used for water flooding. Cathodic protection and use of corrosion-resistant materials have practically stopped corrosion in shallow-water-source wells. Limited tests have failed to disclose a completely satisfactory inhibitor but use of inhibitors appears to be best method for protecting injection wells. Test data are given. Photos, diagram, tables.—INCO. 13255

4.6.4
Water Treatment Problems in Open-Type Recirculating Cooling Systems. G. C. PAULSEN, JR. Magnus Chem. Co., Inc. *National Engr.*, **60,** No. 8, 26-27, 39 (1956) August.
Maintenance factors that must be considered for satisfactory operation and protection of equipment include corrosion of metal, scale deposition, algae growth, chemical effects on wooden cooling towers and controls for total solids concentration. Inorganic or organic type inhibitors are commonly used for protecting metal surfaces. Use of sequestering agents and dispersants in controlling scale formation is discussed. Control of algae, pH and total solids concentration is described. Since a package treatment designed for the average plant cannot be expected to serve in all plants, operating efficiency and maintenance demand careful engineering control and tailor-made treatment.—INCO. 12837

4.7 Molten Metals and Fused Compounds

4.7, 6.2.5
Dynamic Corrosion Test of Type-347 Stainless Steel in Sodium. N. G. MILLS AND R. F. KOENIG. Knolls Atomic Power Lab. U. S. Atomic Energy Comm. Pubn., KAPL-M-NGN-1, October, 1950 (Declassified February 9, 1956), 9 pp. Available from Office of Technical Services, Washington, D. C.
As part of the program to compare various sodium purification treatments, a six-month dynamic corrosion test employing thermal circulation was made with aged and filtered sodium to determine the corrosion and erosion resistance of Type 347 stainless steel. The temperature relationships were observed for indications of plugging by metal or oxide deposition. No plugging was encountered although there appeared to be some iron deposited immediately downstream from the cooling fins and in the annulus around the specimen. The results showed that Type 347 stainless steel was very resistant to corrosion and erosion in aged and filtered sodium at 500 C (932 F). It was also shown that such a system can be operated with moderate flow rates (0.3 ft./sec.) and relatively small openings (3/16 inch) without difficulty. (auth).—NSA. 13271

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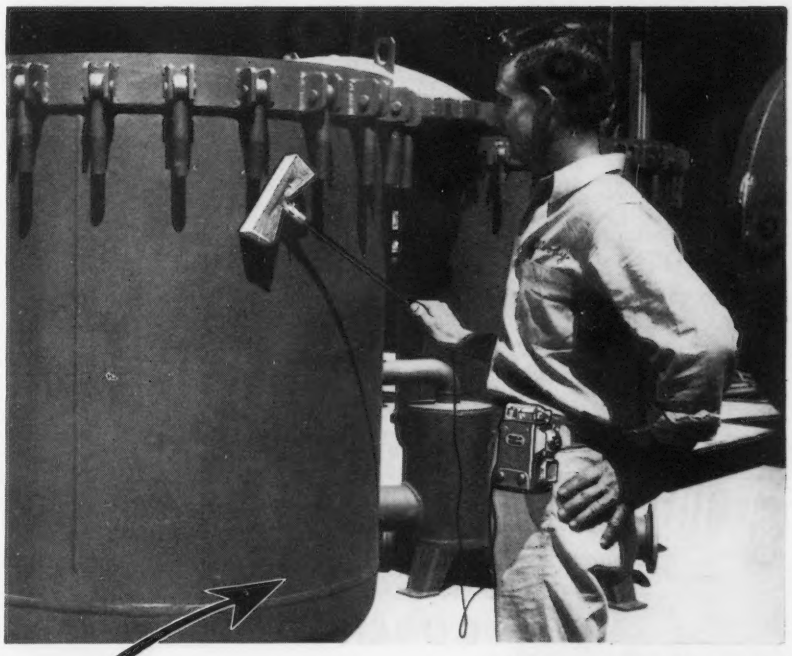

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4.7, 6.3.10

The Action of Sodium Hydroxide Melts on Alloys of Nickel, Molybdenum, and Iron at 815 C. G. P. SMITH AND E. E. HOFFMAN. Oak Ridge National Laboratory. U. S. Atomic Energy Commission Pubn., ORNL-2131, November, 1956, 7 pp.

Reaction mechanism involves selective leaching of iron and molybdenum from solid solution with nickel and a resultant formation of subsurface porosity. Alloys containing at least 70% nickel showed more resistance to corrosion than others tested.—MR. 13348

4.7, 6.3.10, 4.3.3

Reactions in Sodium Hydroxide-Nickel System. D. M. MATHEWS AND R. F. KRUH. Univ. Arkansas. *Ind. & Eng. Chem.*, **49**, No. 1, 55-58 (1957) Jan.

Current interest in high temperature systems led to attempt to establish course of reaction between a metal and a hydroxide at temperatures up to 1000 C. Reactions between sodium hydroxide and nickel, carried on in vacuum at temperatures near 1000 C produced hydrogen, water, sodium oxide, nickel oxide, sodium nickelate and sodium. All reactions are driven by removal of a gaseous product and in a closed system no net reaction occurs. Substantial attack of nickel by sodium hydroxide in vacuum begins at about 600 C. There is no evidence that a steady hydrogen pressure is attained at a given temperature. Molten sodium hydroxide creeps along surface of vessel containing it and is a source of great experimental difficulty. Compound Na_2NiO_2 occurs as clear green crystals of needle-like habit and is rapidly hydrolyzed to sodium hydroxide and nickel hydroxide. Graphs, tables. —INCO. 13191

4.7, 6.3.20

Corrosion of Zirconium in 1000 F Sodium (Memo Report No. 110). S. J. RODGERS AND J. W. MAUSTELLER. Mine Safety Appliances Co., Callery, Pennsylvania, Contract NObs-65426, April 23, 1956, 5 pp.

The corrosion of zirconium was studied as a function of oxygen concentration in 1000 F sodium flowing at 3 fps in a nickel system. The corrosion rate varied from 0.8 mg./cm.²-mo. at 0.005 wt.% oxygen to 1.0 mg./cm.²-mo. at 0.036 wt.% oxygen. Samples were covered with a black film and all showed weight gains. (auth).—NSA. 13360

4.7, 6.3.20

Use of Zirconium in Liquid-Sodium Systems. F. E. BOWMAN AND D. D. CUBICCIOTTI. Atomics International, Canoga Park, California. *A.I.Ch.E. Journal*, **2**, 173-176 (1956) June.

The attractive nuclear properties of zirconium make it a highly desirable core material for sodium-cooled reactors. The elevated temperature strength while low is sufficient for certain applications. Development of higher strength alloys is underway. Sodium in itself is completely compatible with zirconium; however, the nonmetallic contaminants, namely oxygen, hydrogen, and nitrogen, can effect serious damage. The primary problem in the use of zirconium in a sodium system, then, lies in controlling these impurities in the sodium. (auth).—NSA. 12751

5. PREVENTIVE MEASURES

5.1 General

5.1

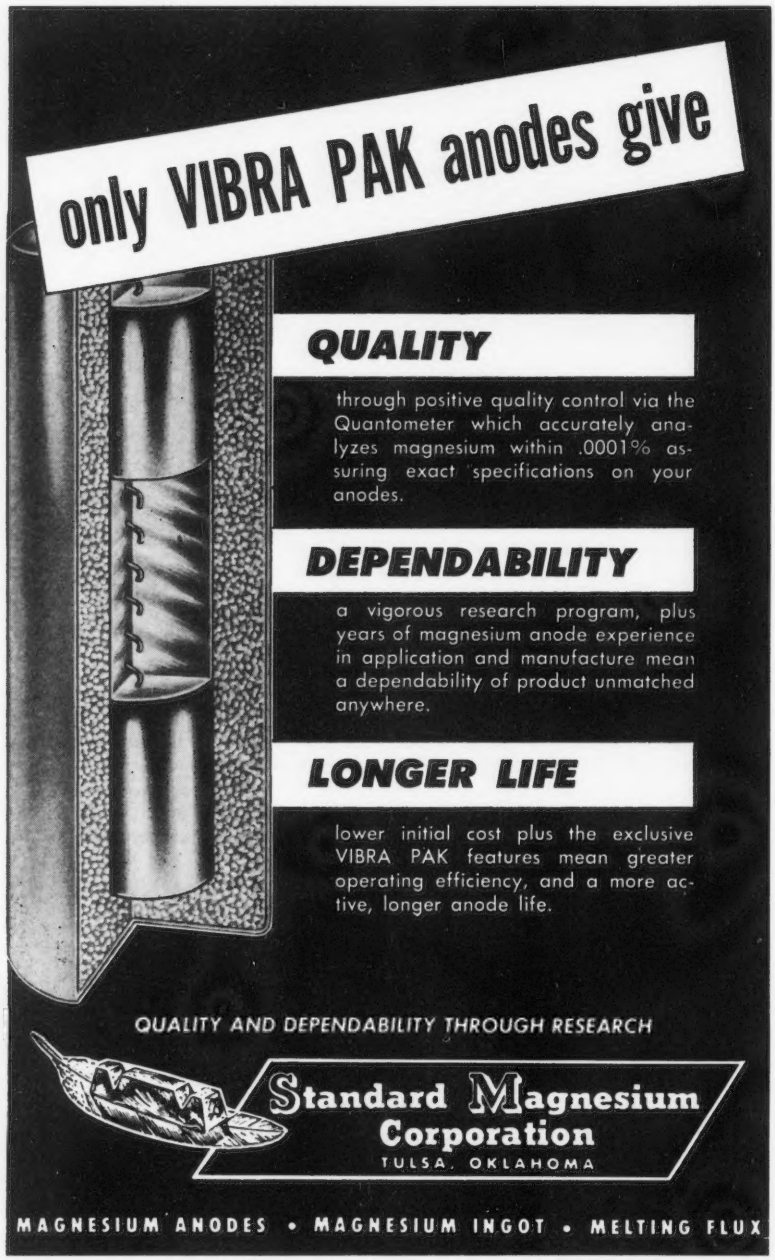
Corrosion Prevention. (In French.) D. PETROCOKINO AND J. FRASCH. *Corrosion et Anticorrosion*, **14**, No. 7, 246-251 (1956) July-August.

Problems of corrosion prevention by cathodic protection, inhibitors and coatings.—MR. 13330

5.1, 6.1, 3.1, 1.6, 8.4.3

Corrosion Control Short Course, April 3-5, 1956. Book, 1956, 238 pp. Available from: Ted V. Sherman, Business & Industrial Services, University of Oklahoma, North Campus, Norman, Oklahoma.

Compiled from papers given at the third annual corrosion control short course sponsored by the NACE Central Oklahoma Section and the College of Engineering and the Extension Div., Univ. of Oklahoma. Papers include: Theory of Corrosion Inhibitors, P. D. Muir; Use of Rectifiers in Corrosion Control Work, R. M. Wainwright; Rectifiers vs Sacrificial Anodes for Cathodic Protection of Pipe Lines, W. E. Huddleston; Operation and Maintenance of a Corrosion Control System, O. W. Everett; Corrosion in Steam and Condensate Systems, George M. Keith; Cathodic Protection of Underground Plant and Yard Equipment, G. L. Doremus; Corrosion Control in Recirculating Cooling Water Systems, P. G. Bird; Practical Answers to Stress-Corrosion Cracking Problems, R. C. Schueler; Economic Aspects of Corrosion Control in Oil and Gas Production, R. C. Buchan; General Economic Evaluation of Corro-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sion Control Programs, K. T. Feldman and R. J. Osborn; Economics of Pipeline Coatings, A. W. Peabody; Planning a Corrosion Control Program for a Gas Distribution System, W. T. Burgress; Field Testing of Inhibitors, Bonner O. Stampley; Internal Coating of Pipelines, K. W. Turner; Instruments for Corrosion Control, G. L. Doremus; Inspecting for Corrosion, D. J. Evans; The Safety Value of a Corrosion Control Program, J. N. Byers; Principles for Selecting Plastics in Combatting Corrosion, H. D. Boggs; Causes and Prevention of Bacterial Corrosion, J. N. Breston; Corrosion in Waterflood, H. L. Bilhartz; Protection of Oil Well Casing, W. C. Koger; The Role of Iron Carbides and Impurities in the Corrosion of Plain Carbon Steels, F. J. Radd, L. H. Crowder and R. L. McGlasson; The History of Carbon Dioxide Corrosion in Oil and Gas Wells, Morris Bock. 12681

5.2 Cathodic Protection

5.2.1

Cathodic Protection. (In Dutch.) J. W. Boon. *Metalen,* **11,** No. 10, 219-226 (1956) May 31.

Principle of cathodic protection. Electrical and electrochemical considerations. Galvanic method. Reactions at the anode and cathode. Criteria of complete protection. Advantages and deleterious secondary effects. Practical results of application.—BTR. 12732

5.2.1, 1.2.5

How Bonding Cables Prevent Arcing. R. I. Stark. Paper before San Joaquin Valley Oil Industry Safety Conference, October 9-10, 1956. *Oil Gas J.,* **54,** No. 85, 157-158 (1956) Dec. 17.

Description of replacement of 40 feet of a high-pressure-gas pipeline and bonding technique used. In a broken pipe wall, the cathodic protection circuit is broken and an arc is drawn that causes fire or explosions. The only positive way to prevent arcing when pipe wall is broken is to provide an alternate patch around break with a bonding cable.—INCO. 13392

5.2.1, 4.6.11

Processes of Formation and Destruction of Films in the Case of Cathodic Protection of Steel Surfaces in Sea Water. I. B. Ulanovskii. *J. Applied Chem., USSR* (Zhur. Priklad. Khim.), **29,** No. 7, 1056-1062 (1956).

The mechanism was studied to effect savings in current consumption. 1 table, 2 figures, 13 references.—ATS. Translation available: Associated Technical Services, P. O. Box 271, East Orange, New Jersey. 12632

5.2.1, 7.7

Corrosion Control System for Underground Cables in Conduit in a Stray Current Metropolitan Area. Edward P. Teale. *Corrosion,* **13,** No. 3, 165t-172t (1957) March.

Several miles of a main conduit route in a metropolitan area containing bare lead sheath telephone cables has been protected satisfactorily from stray current corrosion by a single 700,000 cm drainage wire. During and following the war period there was a rapid expansion of all types of underground utilities throughout the area. This altered the subsurface network conditions, causing

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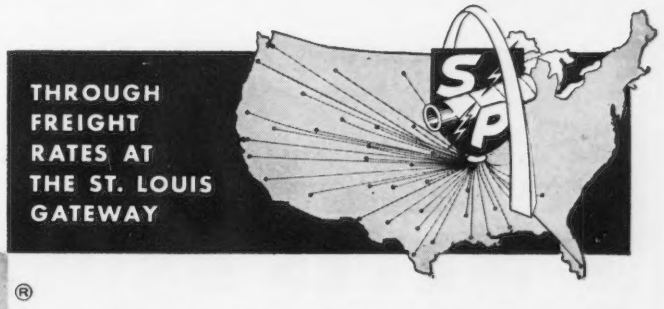

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the efficiency of the former protection to be greatly diminished.

Investigation indicated that in order to reestablish adequate protection by a single natural drainage connection, a much larger wire would be required. Further investigation indicated that by utilizing natural drainage from two points on the conduit system, thus providing the equivalent of distributed drainage, the size of wire for a natural drainage connection could be reduced appreciably.

As the result of additional study it was decided to utilize a controlled type of rectifier employing two stages of magnetic amplification. The primary purposes of such equipment were to overcome the IR drop in the drainage conductor and to limit the drainage current to the minimum value required by the natural potential difference between cable and earth. Employment of this equipment permitted the use of a distributed drainage arrangement, utilizing the original drainage facilities for one branch and a much smaller wire for the second branch.

This paper describes the system in detail, discusses the results obtained and the problems encountered in adjusting the rectifier equipment to obtain the desired operating characteristics. 13130

5.2.1, 7.7, 6.3.8

The Protection of Cable Sheathing: Cathodic Protection of Telecommunication Cables. J. GERRARD AND J. R. WATTERS. Paper before Soc. Chem. Ind., Corrosion Group, Symp. on Protection of Cable Sheathing, London, Nov. 18, 1955. *Chemistry & Industry,* No. 40, 1060-1068 (1956) Oct. 13; disc., *ibid.,* No. 42, 1196-1213 (1956) Oct. 27.

Causes of corrosion of lead cable sheathing include dissimilar metal corrosion, electrolysis caused by currents generated in lead sheath and natural earth currents. Experimental application by the GPO of cathodic protection at 172 sites, mostly to sheathed cables laid in earthware ducts, is discussed. Impressed current system is generally employed using a ground bed situated close to the exchange; gives adequate protection for a two mile radius round the exchange for current less than two amperes. Assessing and methods for reducing possibility of corrosion damage to other buried structures: in this connection reference is made to the work of the "Joint Committee for the Co-ordination of Cathodic Protection of Buried Structures." 13215

5.2.1, 8.9.3

Distribution of Potential Along Piping Using Cathodic Protection. (In Russian.) N. A. TSEKUN. *Energeticheskii Biulleten',* No. 10, 13-16 (1956) Oct.

A study of the divergence between theoretical calculations and practical data. Determination of the necessary corrections for existing formulas.—BTR. 13420

5.2.1, 8.9.3, 1.2.2

A 17-Year Cathodic Protection Program Has Paid Off. G. R. OLSON. United Gas Pipe Line Co. *Refining Engr.,* **28,** No. 13, D35-D36, D38-D40, D42-D43 (1956) December.

Discussion of cost and results from application of cathodic protection for control of external corrosion on United Gas Pipe Line Company's system during a 17 year period. Graphs, 13 references.—INCO. 13308

5.2.2

Investigation of the Effectiveness of Cathodic Protectors Made of Aluminum-Zinc Alloys. (In Russian.) V. V. GERASIMOV AND I. L. ROZENFELD.) *Bull. Acad. Sci., URSS,* Classe Sci Chim., No. 9, 1050-1052 (1956) Sept.

Investigation of stationary electrode potentials, magnitude of protective current, anodic polarization and stability of six alloys.—BTR. 13200

5.2.2

On the Characteristics of Aluminum-Zinc Galvanic Anode. H. SHIGENO AND H. TOGANO. *Repts. Govt. Chem. Ind. Res. Inst.,* Tokyo, **1,** No. 10, 373-380 (1956) October.

The effects of current densities (0.05-3 mA/cm²) and anode compositions (4, 6, 8, 10, 19 and 65% zinc) on aluminum-zinc anode characteristics in sea water have been quantitatively investigated.

The principal conclusions derived from this work are as follows: 1) The anode potentials of aluminum-zinc alloys (4-19% zinc) range from about —950 to 960 mV referred to a saturated calomel electrode. For these alloys, anodic polarization is apparently little perceived. On the other hand, 65% zinc alloys show an anodic potential of about —1000 mV, but exhibit some anodic polarization. 2) In quantity of current passed per weight loss, aluminum-zinc alloys (4-19% zinc) are superior to both magnesium anodes and zinc anodes; this superiority is the principal merit of aluminum-zinc galvanic anodes. For 65% zinc alloy the quantity is small, therefore it does not become an object of practical use. 3) Pitting corrosion is notable with every aluminum-zinc alloy tested, but the existence of preferential corrosion between different metallic structures (for example, α and β) is not distinct. 4) As a constituent, high purity aluminum can not much improve the anode performance of these alloys.—JSPS. 12971

5.2.2

Aluminum-Zinc Galvanic Anode. H. TOGANO. *Corrosion Engineering,* **5,** No. 4, 191-194 (1956) August.

Performance characteristics of aluminum-zinc galvanic anode in artificial sea water have been studied in the laboratory. Effects on anode performance of current densities (0.05-3 mA/cm²) and alloy compositions (4, 6, 8, 10, 19 and 65% zinc) have been investigated quantitatively.

Principal conclusions derived are:

1) Anode potentials of aluminum-zinc alloys (4-19% zinc) measure about —950 to —960 mV (referred to sat'd. calomel electrode).

2) Anodic polarization, as affected by time or current density, is scarcely recognized for these alloys (4-19% zinc).

3) In quantity of current per gram, these alloys are superior to magnesium and zinc anodes. In current density under 1 mA/cm², aluminum-4% zinc alloy shows the highest value.

4) Pitting corrosion is notable and this non-uniform anode consumption is a great fault.—JSPS. 13101

5.2.2, 1.6

Behavior of the Reactive Anodes in Magnesium and in Zinc. (In French.) J. VAN MUYLDER AND M. POURBAIX. Centre Belge d'Etude de la Corrosion, Rapport Technique No. 34, May, 1956, 59 pp.

Theoretical consideration of cathodic protection by reactive anodes, using pH tension diagrams. Relationship between polarization curves and anodic production of current. Influence of metals, medium and density of current on anodes. Practical applications. 22 graphs.—MR. 13406

5.2.2, 7.2, 8.4.3

How Magnesium Anodes Retard Casing Corrosion. H. A. BRADY. Corrosion Services Inc. *World Oil,* **144,** No. 1, 160-162 (1957) Jan.

Briefly reviews principal causes of casing corrosion and conditions warranting use of magnesium anodes. Determination of current needed for cathodic protection and planning of anode installations are discussed. Length of casing protected in cases cited varies from 5000 to 6500

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feet with 6-9 anodes at each well. Diagram, graphs.—INCO. 13182

5.2.2, 7.6.4

Checking Corrosion in Galvanized Iron. R. IRONMAN. *Modern Power & Eng.*, **50**, No. 11, 84-86 (1956) Nov.

Description of causes and prevention of corrosion of galvanized iron hot water tanks by magnesium anode cathodic protection. Photos.—INCO. 13178

5.2.2, 8.9.5

Cathodic Protection of an Active Ship Using Zinc Anodes. B. H. TYTELL AND H. S. PREISER. *J. Am. Soc. Naval Engrs.*, **68**, 701-704 (1956) Nov.

High-purity zinc provided excellent protection; a useful life of four or five years is anticipated.—BTR. 13418

5.2.3, 5.4.10, 8.9.3

Cathodic Protection for Pipeline Across Arabian Desert. K. R. WRIGHT. Arabian Am. Oil Co. *Civil Eng.*, **26**, No. 5, 33-36 (1956) May.

The Trans Arabian Pipeline from Quatif in the Persian Gulf to Sidon on the Mediterranean is made of steel protected at buried parts with a coating consisting of asphaltic primer, a coat of asphalt, a single wrap of glass floss, another coat of asphalt and a wrap of Perrault padding. Rectifiers of the cathodic protection installation were unable to give proper protection to the line so welding generators were utilized on a temporary basis at intermediate points to supply electrical current to accompanying beds. Method employed was to make the soil-to-pipe electrical potential large enough to stop electron flow from the pipe to the earth. An electrical current was supplied to the soil either through graphite or scrap-steel anodes which traveled through the earth into the pipeline wherever a defect in the protective coating occurred. To complete the electrical circuit the current traveled along the pipe to a lead running back to the generator supplying the current. Design problems and desert construction methods are discussed. Illustrations.—INCO. 12654

5.2.3, 8.2.2, 3.3.2

The Application of Cathodic Protection to Sea-Water Intake Structures of Power Plants. S. TAKESHIMA AND J. OYAMA. *Corrosion Engineering*, **5**, No. 1, 33-36 (1956) Feb.

A cathodic protection system has been installed on the bar screens and travelling screens in Amagasaki First Power Plant and newly-installed Himeji Power Plant, both run by the Kansai Electric Power Co., Ltd. and in the Chiba Steel Mill of the Kawasaki Steel Manufacturing Corporation. Direct currents from selenium rectifiers were applied with magnetite anodes at the rate of 0.129-0.33 amp. per sq. meter of protected surface.

For these screens, the average potential to earth was kept to 1,000 millivolts (referred to copper sulfate half-cell).

It was verified by measurements of screen potential and by observation that the results obtained were satisfactory. As to the fouling of screen surface by marine shells and sea-weeds, several instances have been observed which reveal that high direct current density prevents their growth.—JSPS. 12621

5.2.3, 8.9.3, 4.5.1

Cathodic Protection of the Coated Steel Gas Main Distribution System in New Orleans. SIDNEY E. TROUARD. *Corrosion*, **13**, No. 3, 151t-161t (1957) March.

A report is given of the practical considerations involved in the cathodic protection of the coated steel gas main distribution system in New Orleans. Topics discussed include topography and soils in New Orleans, types of gas mains used in system, use of coatings, use of galvanic anodes; experience with rectifiers and ground beds, use of insulated joints, importance of educational program and cost of cathodic protection. Data are given for a portion of one periodic rectifier survey and yearly tests on three galvanic anodes.

Results secured were good. It was found that successful operation of cathodic protection in New Orleans could be materially aided by observing the following procedure: 1) Secure reasonably good coating of the mains to be protected, 2) Maintain the systems to be protected electrically continuous, and free from electrical contact with other metallic subsurface structures, 3) Maintain the electrical potential of the systems to be protected at least —0.85 volt to a copper sulfate reference electrode placed in the earth, 4) Maintain continuous operation of the device furnishing the current for cathodic protection, 5) In general use many small (rather than a few very large) units for furnishing the cathodic protection currents. This minimizes problems of cathodic coordination. 13137

5.2.4, 3.4.6, 3.6.2

The Effect of Dissolved Oxygen on Corrosion of Steel and on Current Required for Cathodic Protection.

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187a

E. SCHASCHL AND G. A. MARSH. *Corrosion*, **13**, No. 4, 243t-251t (1957) April.

The interrelationship of corrosion rate of steel, dissolved oxygen concentration, agitation, electrode potential and cathodic protection current density were studied under controlled laboratory conditions. While the corrosion rate of steel is essentially linear with amount of dissolved oxygen, the rate appears to fall almost to zero near 1.0 ppm dissolved oxygen rather than at zero ppm dissolved oxygen as commonly assumed. In the region below 2 ppm dissolved oxygen, there is almost no effect of agitation on corrosion rate; above 2 ppm there is a large effect. By measuring the corrosion rate of an unprotected specimen, it should be possible to compute the minimum current density needed for cathodic protection in the field.

In acidic environments, less current is needed for protection than is predicted by analysis of the mechanism of cathodic protection. This fact suggests that the anodic reaction in low pH corrosion proceeds by the removal of "chunks" of metal which contain several atoms. The "chunk effect" also appears to operate in the neutral range, but only when the corrosion rate is very high.

The effect of differential aeration on corrosion rates is discussed in detail. 13441

5.3 Metallic Coatings

5.3.2

Steel Corrosion Prevention by Metallic Coating and Especially by Hot Galvanizing. (In French.) J. F. H. VAN EIJNSBERGEN. Centre Belge d'Etude de la Corrosion, Rapport Technique No. 36, July, 1956, 3-7; disc., 7-8. Available from CEBELCOR, 21 rue des Drapiers, Brussels, Belgium.

Zinc, aluminum or cadmium coatings, methods used and influence of zinc thickness on capacity to withstand corrosion. —MR. 12642

5.3.2

Bright Anodized Aluminum Versus Nickel-Chromium Plating: Debate before Inst. Metal Finishing, London. *Metal Finishing J.*, **2**, No. 22, 357-363, 389 (1956) October.

Debate was held on motion that bright anodized aluminum will replace nickel and chromium plating. Speaking for motion were I. Walker and E. E. Spillett; H. C. Castell and T. E. Such spoke in favor of nickel-chromium plating. Topics touched upon included reflectivity, life of surface finish after atmospheric exposure, comparison of corrosion products, relative abrasion resistance, cost factors, rigidity and durability of bright anodized aluminum and nickel-chromium-electroplates. Most references were made to applications in the automotive industry. Motion was rejected in favor of nickel.—INCO. 13275

5.3.2

Selecting Electroplates for Iron Powder Parts. W. H. SAFRANEK AND M. W. WIRTH. *Materials & Methods*, **44**, No. 5, 104-109 (1956) Nov.

Based on a comprehensive investigation, article gives specific recommendations on bath formulations and detailed data on appearance, thickness and uniformity, salt spray resistance and humidity resistance for cadmium, zinc, nickel, chromium and copper electroplates on iron powder parts. Nickel baths listed are Watts-type nickel sulfate and nickel sulfamate baths; latter deposits nickel with better efficiency and less hydrogen gassing. Photomicrographs compare structures of various electroplates. High and low density and impregnated parts plated with nickel, chromium and copper (sulfate bath) are equal in salt spray resistance. Parts plated in nickel sulfamate bath exhibited least corrosion during humidity tests. Tables.—INCO. 13345

5.3.2

Selecting Wear-Resistant Materials. W. L. LUTES AND H. F. REID, JR. The McKay Co. *Chem. Eng.*, **63**, No. 6, 243-246 (1956) June.

Discusses trend toward standardization in choice of hard-facing alloys, as manifested in proposed AWS-ASTM specifications and reviews factors to be considered in selecting wear-resistant materials for chemical process equipment. Discussion is limited to iron-base alloys while chart with comparative ratings of hard-surfacing alloys includes data for cobalt-base, carbides, copper-base and nickel-base (Monel, Nichrome, Hastelloy) alloys. Hardness, general atmospheric corrosion resistance, resistance to earth abrasion, resistance to hot deformation, service under heavy abrasion and service in sliding and rolling wear are evaluated in chart. Eroding, grinding, gouging and frictional types of abrasion and materials best suited for each type are considered. Applications discussed are equipment for movement and storage of liquids, equipment for handling dry materials, grinding, mixing and crushing equipment and high temperature service application.—INCO. 12763

5.3.2

Limitations of Plated Nickel in Jet Engine Design. R. W. MOELLER AND W. A. SNELL. Paper before Am. Electroplaters' Soc., 43rd Ann. Conv., Washington, D. C., June 17-22, 1956. *Tech. Proc. AES*, **1956**, 230-233.

There are four principal limitations to use of plated nickel in jet engine design: degree of corrosion resistance available from deposits thin enough for dimensional tolerance (overcome by use of composite plate of diffused nickel-cadmium); residual tensile stress and consequent reduction in fatigue strength (overcome by use of low stress solutions, proper heating after plating and chemical cleanliness); processing and control difficulties on production scale application; and specialized drawbacks such as brittleness of electroless nickel plated parts (solution lies in development of deposits with greater ductility at operating temperatures designated). Data are presented for residual stress in nickel and nickel-cadmium plates (from Watts' type and sulfamate baths) correlated with endurance limit and for 100 hour salt spray test for nickel plate and nickel-cadmium diffusion plate. Photomacrographs show crack pattern and fracture in electroless nickel plate. Graphs.—INCO. 13263

5.3.4

Effect of Baking on Delayed Fracture of Electroplated Ultra High-Strength Steel. B. F. BROWN. Naval Research Laboratory, October, 1956, 20 pp. Available from: Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 121499).

Evaluated in this report is the effectiveness of baking treatments on the susceptibility of plated high-strength steel to failure by delayed fracture. Various methods used to test electroplating embrittlement are discussed. Delayed fracture tests on notched tensile specimens were conducted to assess the relief of the embrittlement in ultra high strength steel by baking at 350F after plating. This relief in chromium-plated steel was essentially complete after baking 24 hours. Baking as long as 200 hours brought incomplete relief in cadmium-plated steel and essentially no relief in zinc-plated steel. To alleviate susceptibility to delayed fracture in cadmium-plated steel, an electroplating bath is proposed which either does not discharge much hydrogen or alternatively gives a plate sufficient porosity to permit exit of hydrogen. 13019

5.3.4

Polarization and Crystal Growth in the Electrocrystallisation of Copper. (In German.) R. SROKA AND H. FISCHER. *Z. Elektrochem.*, **60**, No. 2, 109-118 (1956).

Investigation of the relation between polarization and form of deposition of copper from acid sulfate solution.—BNF. 13060

5.3.4

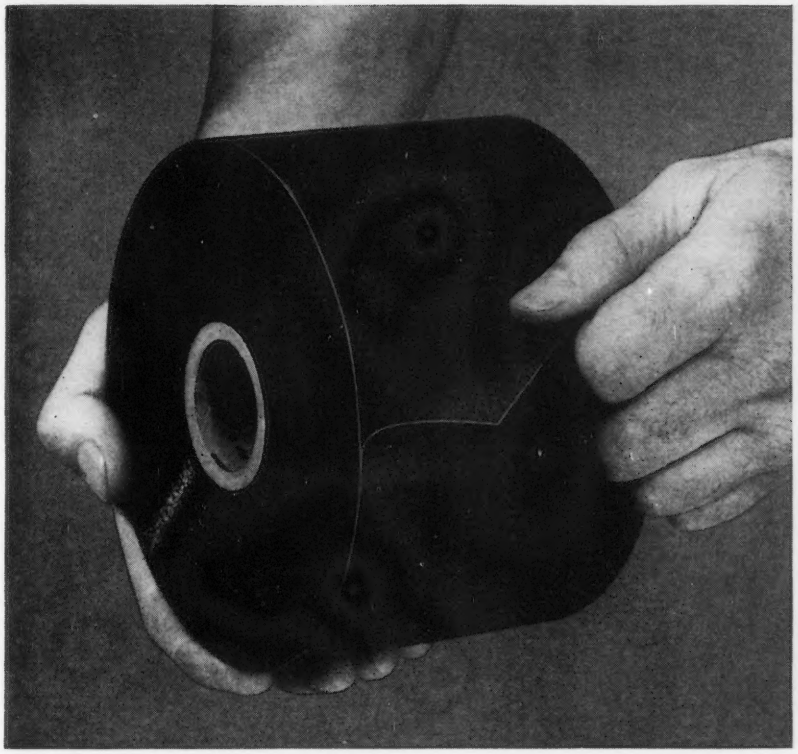
Some Experimental and Practical Aspects of Heavy Rhodium Plating. F. H. REID. Paper before Inst. Metal Finishing, London, Oct. 17, 1955. *Bull. Inst. Metal Finishing* (Trans. IMF), **6**, No. 2, 107-142 (1956) Summer.

Results of laboratory studies on various types of acid rhodium plating electrolytes, with particular reference to sulfate and phosphate-sulfate solutions and including remarks on phosphate, fluoborate and sulfamate electrolytes. Deposits from sulfate and phosphate-sulfate solutions are examined with reference to thickness, adhesion, surface condition, cracking incidence, internal stress and hardness. Effects of processing variables on electrolyte characteristics and influence of composition and deposition factors on internal stress are discussed. Factors influencing cracking of rhodium deposits are considered and recommendations are made concerning formulation of sulfate electrolytes and optimum operating conditions for 0.0002-0.002 inch thick rhodium deposits. Detailed procedures are given for direct rhodium and silver/rhodium plating of common basis metals (silver, nickel, copper, nickel silver, brass, beryllium-copper and phosphorus bronze). Tables, photomicrographs.—INCO. 12893

5.3.4

Electrolytic Tin-Plating of Steel Sheet from Chloride Solutions. (In Russian.) V. P. KOCHERGIN, T. A. NIMVITSKAYA AND M. YA. V'YUNOVA. J. Applied Chem., USSR *(Zhur. Priklad. Khim.)*, **29**, No. 1, 59-63 (1956).

The direct compensation method [Loshkarev *et al., Zhur. Fiz. Khim.*, **9**, 1412 (1939)] was used to investigate the effect of additions of phenol, α- and β-naphthol, phenolsulfonic acid, α-naphthylamine, thioguanidine, ρ-toluidine, α-aminopyridine, ρ-aminophenol, diphenylamine, isopropylamine, ethylenediamine, urotropine and camphor on the polariza-



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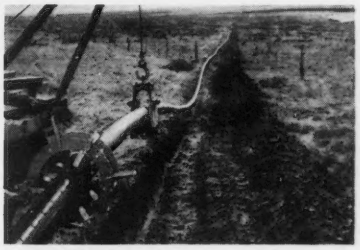

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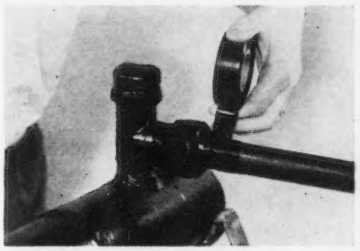

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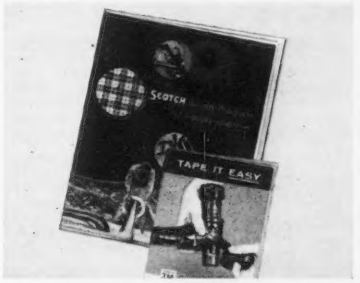

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tion curves for baths containing either stannous chloride 25, sodium chloride 5, hydrochloric acid 10, glue 2-3 g/l, or stannous chloride 50, sodium chloride 10, hydrochloric acid 10, glue 2-3 g/l, at 20 C. Additions of phenol or α- β-naphthol, unlike the amino-compounds, caused an appreciable increase in the cathode potential. The tin deposits from the chloride deposits were dendritic or spongy, but on simultaneous addition of phenol and α- or β-naphthol compact deposits were obtained. The deposits were flow-brightened in a vertical tube furnace at 650-700C, using a flux containing zinc chloride 50, ammonium chloride 16 g/l, and <1 sec. after the appearance of the "mirror" were quenched in water; on the majority of the specimens drops of various sizes were formed. The formation of drops increased with increasing deposit thickness, and was also more noticeable with dendritic or spongy deposits. However, it could be prevented by first treating the cold-rolled steel sheet for 1-2 sec. in a bath containing $Na_2SnO_3 \cdot 3H_2O$ 110, sodium hydroxide 10, CH_3COONa 10 g/l at 75C and 1-2 amp./dm.² before deposition from the chloride bath. The corrosion-resistance of the tinplate will be best when the $FeSn_2$ content of the coating is least, i.e. when the height of the "mirror" of fused tin is a minimum. The recommended plating process is 1-2 sec. at 75C and 1-2 amp./dm.² in a bath containing $Na_2SnO_3 \cdot 3H_2O$ 45-55, sodium hydroxide 9-11.5 g/l, followed by deposition at 15-20 amp./dm.² and 20C from a bath containing stannous chloride 50-57, sodium chloride 10, hydrochloric acid 12-18, glue 3, phenol 20, α-naphthol 0.1 g/l.—MA. 13041

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5.3.4

Deposition of Titanium from Titanium-Oxygen Alloys on Copper, Iron and Mild Steel. S. T. SHIH, M. E. STRAUMANIS AND A. W. SCHLECHTEN. *J. Electrochem. Soc.*, **103,** No. 7, 395-400 (1956) July.

Thick corrosion resistant coatings of titanium on ingot iron, mild steel and other metals were obtained by embedding them in a mixture of sodium chloride or potassium chloride and a titanium-oxygen alloy (high in titanium content), and heating in helium for several hours (at 850C for copper, 1000C for iron). Thickness of coating depends primarily on temperature, also on time, oxygen content of the alloy, and composition of bath. Another account (In German): *Metall,* **10,** No. 19/20, 901-909 (1956) October.—BNF. 12953

5.3.4

Plating on Unusual Metals. C. L. FAUST AND J. G. BEACH. *Plating,* **43,** No. 9, 1134-1142 (1956) September.

Discusses cladding by electroplating for such high temperature structural and nuclear reactor materials as aluminum, beryllium, molybdenum, niobium, titanium and zirconium, their alloys and alloys such as Inconel and Stellite. Techniques of activation and plating beryllium, zirconium, niobium, molybdenum, bismuth and Inconel are summarized. Compositions and deposition data are presented for aluminum, bismuth, copper, iron, nickel and zinc plating baths. Neutron capture ability of nickel limits its thickness as coating to about 0.005-inch. Iron shows less neutron capture, costs less and is more available than nickel; it is considered wherever lower oxidation resistance is not a handicap. Photomicrographs show alternate layers of bismuth and nickel on aluminum, nickel- and iron-plated niobium and nickel- and iron-plated beryllium. Tables give adherence data for nickel on zirconium. Alloying by diffusion at the interface of cladding-basis metal is discussed.—INCO. 12815

5.3.4

Less Common Metals and Alloys: Electrodeposition from Aqueous Solution. M. L. HOLT. *Metal Finishing,* **54,** No. 9, 48-55 (1956) Sept.

Review article on electrodeposition of titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium alloys, molybdenum, tungsten, manganese and rhenium. 35 references.—BNF. 12798

5.3.4

The Electrodeposition of Rhodium. J. FISCHER AND F. LEONHARD. *Metall,* **10,** 608-617 (1956) July.

Notes on bath compositions, operating conditions, effect of impurities in the bath, decorative and heavy rhodium deposits, corrosion-resistance of rhodium deposits, structure and effects of bath, compositions and heat-treatment are given (reference to private communication by E. C. Rhodes).—INCO. 12796

5.3.4

The Inhibiting Action of Aluminium in Hot Dip Galvanizing Baths on the Formation of Iron-Zinc Alloy Layer. (In German.) D. HORSTMANN. *Arch. Eisenhuttenw.,* **27,** No. 5, 297-302 (1956) May.

It is suggested that during the attack by aluminum-containing zinc melts on iron surfaces an Al_8Fe_2 layer is formed which exercises a strong inhibiting influence. This inhibition breaks down after a certain time, depending on the aluminum content and the temperature of the zinc bath. Subsequently a strong attack by zinc occurs, which after long immersion times lessens slowly.—BNF. 12785

5.3.4

The Effect of Ultrasonics on Electrodeposition of Metals. A. ROLL. *Metalloberflache,* **10,** 230-233 (1956) August.

Review of known effects of ultrasonics, supplemented by new data, with particular reference to nickel- and chromium-plating.—INCO. 12916

5.3.4

A Study of Whisker Formation in the Electrodeposition of Copper. P. A. VAN DER MEULEN AND H. V. LINDSTROM. *J. Electrochem. Soc.,* **103,** No. 7, 390-395 (1956) July.

Under certain conditions the electrolysis of acid cupric sulfate using copper electrodes leads to formation of whiskers of copper on cathode. Certain additives (cystine, thiourea, Safranine T, phenazine and other organic compounds) in electrolyte are capable of overcoming tendency toward abnormal growth and act to give smooth copper deposits. Possible mechanisms involved in electrolytic copper filament formation and its prevention are discussed; hypothesis to explain process is suggested.—BNF. 13102

5.3.4, 5.9.4

Hot Dip Galvanizing, 1946-1956, Parts I, II. M. L. HUGHES. *Product Finishing,* **9,** Nos. 7, 8, 181-193, 212; 61-67 (1956) July, August.

Part I. Preparatory treatments (degreasing, pickling, rinsing and fluxing). The hot dipping operation (handling, kettles and heating methods, iron-zinc interaction, bath composition, formation of residuals, zinc recovery).

Part II. Galvanizing of sheet, strip, wire and tube. Based on 281 references, systematically arranged under various headings and sub-headings.—BNF. 13104

5.3.4, 6.3.20

Electrocladding of Zirconium with Platinum. ARCH B. TRIPLER, JR., JOHN G. BEACH AND CHARLES L. FAUST. Battelle Memorial Institute. U. S. Atomic Energy Comm. Pubn., BMI-1097, June 21, 1956, 12 pp. Available from: Office of Technical Services, Washington, D. C.

A method, which included the use of periodic reverse plating, was developed for electrocladding zirconium with sound, porefree platinium coatings up to 1.5 mils in thickness. The method was used to electroclad zirconium specimens with 0.5 to 0.75 mil of platinum for in-pile corrosion tests at ORNL. The in-pile tests were not completed at the time this report was prepared. (auth)—NSA. 13120

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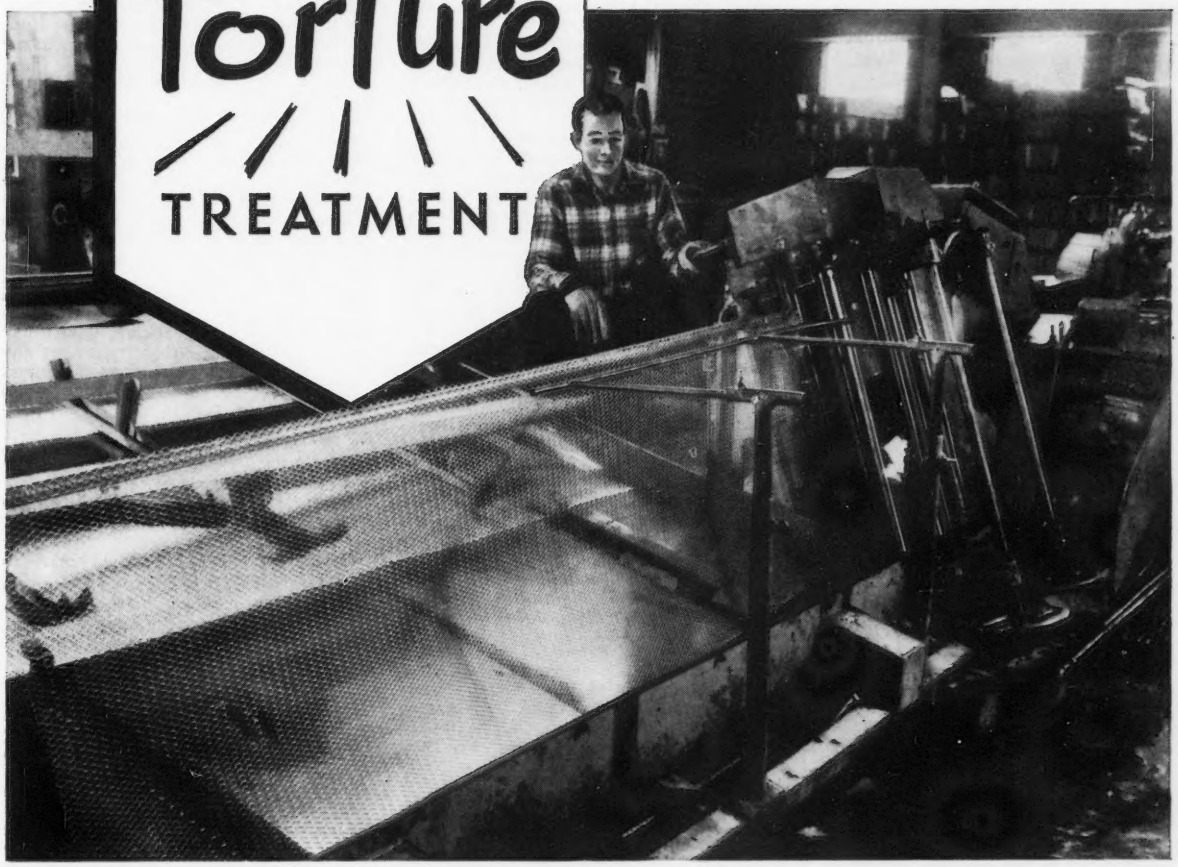

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6. MATERIALS OF CONSTRUCTION

6.2 Ferrous Metals and Alloys

6.2.2

Spheroidal Graphite Cast Iron: A Review. H. MUHLBERGER. *Z. Ver. Deut. Ing.*, **98**, 1449-1461 (1956) August 21.

Review covering production of spheroidal graphite iron (photographs of equipment for introducing magnesium into melt), physical and mechanical properties, heat-treatment, joining methods, surface treatment, machining, bearing properties, damping wear-resistance, design considerations and possible and existing applications.—INCO. 12846

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6.2.3

Low-Temperature Transition of Normalized Carbon-Manganese Steels. T. N. ARMSTRONG AND W. L. WARNER. Paper before Am. Soc. Testing Materials, 58th Ann. Mtg., Symp. on Impact Testing, Atlantic City, June 27, 1955. ASTM Special Technical Publication No. 176, 1956, 40-58.

Determination of energy-temperature relationships for number of carbon-manganese steels as phase of study of effects of welding transition temperature. Steels were divided into five groups according to carbon content (0.04-0.45) and three groups according to manganese content (0.4-1.0) for purposes of comparison. Both keyhole and V-notch Charpy specimens were tested at successively lower temperatures until decided embrittlement was encountered. Energy absorbed in breaking specimen, test temperature and fracture appearance data were used to plot energy-temperature transition curves and fracture-temperature transition curves. Effects of manganese and carbon and of grain size on ductility transition and fracture transition are discussed. Linear relationship between energy absorbed and lateral contraction measured at base of notch holds for these tests. Tables, graphs.—INCO. 13031

6.2.4

Study of 7% and 8% Chromium Creep-Resisting Steels for Use in Steam Power Plant. M. G. GEMMILL, H. HUGHES, J. D. MURRAY, F. B. PICKERING AND K. W. ANDREWS. *J Iron Steel Inst.*, **184**, Pt. 2, 120-144 (1956) Oct.

Begins with review of preliminary development work on various 7% chromium base steels of duplex constitution. Effect of tempering and high temperature properties are discussed for a 7% chromium-3% molybdenum-½% molybdenum steel. Second section deals with systematic study of metallurgical characteristics of various 8% chromium-3% molybdenum steels, ferritic at all times. Constituents present (titanium carbide, titanium nitride, 'Y' phase, Fe_2Ti Laves phase and carbide $M_{23}C_6$ or k carbide), lattice parameter measurement, hardness measurements, effect of rates of cooling and isothermal heating on precipitation behavior and effect of prior structure on creep properties were studied. Discussion covers co-existence of titanium carbide and $M_{23}C_6$, the solubility of Fe_2Ti and $M_{23}C_6$, precipitation of $M_{23}C_6$, solid solution hardening by titanium and the effects of niobium, tungsten, vanadium and aluminum. In final section, high temperature properties of an 8% chromium-molybdenum-titanium steel are outlined in relation to those of 2¼% chromium-molybdenum ferritic and 18/21/1 austenitic creep resisting steel. Tables graphs, photomicrographs.—INCO. 12809

6.2.5, 3.5.8

Nature of Stress Corrosion Cracking of Stainless Steels When Other Types of Corrosion Are Present. W. G. RENSHAW. Allegheny Ludlum Steel Corp. *Corrosion*, **12**, No. 10, 17-18 (1956) Oct.

While phenomenon of stress corrosion in austenitic chromium-nickel stainless steels is characterized by transgranular cracking, certain other kinds of localized attack may be associated with this phenomenon and may influence susceptibility. Cases cited are intergranular and stress corrosion cracking in Type 316 where intergranular cracks have tended to act as notches or starting points; stress corrosion failure in presence of precipitated carbides in Type 317; and pitting in carbide free material which leads to stress corrosion cracks. Photomicrographs.—INCO. 13003

6.2.5, 3.5.8

The Stress-Corrosion Cracking of Austenitic Stainless Steels. Pt. II. Fully Softened, Strain-Hardened, and Refrigerated Material. J. G. HINES AND T. P. HOAR. *J. Iron Steel Inst.*, **184**, Pt. 2, 166-172 (1956) Oct.

Fully softened 18-8, 18-8-titanium, and 18-8-3 molybdenum stressed in tension and exposed to aqueous magnesium chloride solution (42 wt.-% magnesium chloride) at 154C gave stress-corrosion cracking similar to that found for same steels in fully softened and slightly cold-worked condition. Plastic strain or subzero treatment applied to fully-softened plain 18-8 steel reduced time to fracture at any particular applied stress and increased density of cracking. Quasi-martensite formed by either treatment provides active surface regions for crack initiation but does not help crack propagation. Graphs, photomicrographs.—INCO. 12787

6.2.5, 3.8.3

Potential Formation Phenomena of Austenitic Chrome-Nickel Steels in Distilled Water and in Neutral Nalogen Salt Solutions. CARL CARIUS. *Arch. Eisenhuttenw.*, **27**, No. 5, 323-335 (1956) May.

Potential measurements were made of three austenitic chromium-nickel stainless steels (containing 4.7 molybdenum) in distilled water, halogen salt solutions and in hydrogen peroxide solutions of different concentrations. Potential measurement in distilled water at room temperature, after corrosion in mixture of nitric acid-hydrofluoric acid-sulfuric acid-water, shows that potential is controlled by solid oxide layers formed by corrosion. The obtained potential must be considered as the oxide potential. Potential values obtained in netural distilled water with different oxygen contents correspond to potentials of oxygen electrode. Their reversibility in pH range 14-4 is demonstrated by qualitative and quantitative methods. Influence on potential position of chlorine ions using sodium chloride solution as electrolyte is demonstrated with time potential curves. In concentrated sodium chloride solutions potential variations connected with local corrosion occur. Stability of steels is not influenced by sodium fluoride solutions with concentrations up to 1 N. Potential measurements of steel, platinum and gold in hydrogen peroxide solutions showed that hydrogen peroxide in contact with steel acts as an oxidizing agent; in contact with platinum or gold it is oxidized to molecular oxygen under catalytic decomposition. Tables, graphs, photomicrographs.—INCO. 12819

6.2.5, 5.11

Using Stainless Steels for Corrosion Resistance. R. E. PARET. *Machine Design*, **28**, No. 21, 114-121 (1956) Oct. 18.

As a guide to designers, article describes kinds of corrosion and types of stainless steel and summarizes considerations to prevent corrosion in design, fabrication and operation of stainless steel products. Corrosion behavior of 200, 300 and 400 Series is reviewed. Definitions are given for galvanic corrosion, passivity, pitting, crevice corrosion, intergranular corrosion, stress corrosion cracking, corrosion fatigue, fretting cor-

rosion and impingement and erosion. Design considerations cover dissimilar metals, crevices and joints, drainage, surface finish, stresses and gage of metal. Fabrication considerations cover joining, welding, heat treatment and passivation. —INCO. 12839

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.10

Past, Present, and Future of High Temperature Nickel Base Alloys. F. L. VERSNYDER. Paper before Massachusetts Inst. of Technology Session on "Behavior of Materials at Elevated Temperatures," Cambridge, July, 1955. *J. Metals* (Trans. AIME), Sec. 1, **8,** No. 10, 1445-1449 (1956) Oct.

Melting, casting, processing and mechanical properties are given. Table gives compositions of Nichrome, Hastelloys, Inconel, Nimonics, Multi-alloy, Refractalloy 26, Udimet 500, M-252, Rex 400, K42 B, Wasp alloy and Inco alloys. Alloying, structure and special manufacturing problems of nickel alloys are discussed. Nickel base super-alloys have an extremely bright future if they can be made into reliable engineering materials capable of being produced in production quantities. Trend towards higher titanium and aluminum is consistent with knowledge that increasing amounts of solute elements improves creep resistance of a solvent and that precipitation of a second phase, if it is adjusted properly, can increase resistance to rupture. Graphs, photomicrographs, 20 references. —INCO. 13097

6.3.10, 3.8.2

Of Nickel Atoms, Ions and Electrons. W. A. WESLEY. Bayonne Tech. Paper No. 218, February 20, 1956; Paper befor Inst. Metal Finishing, 3rd Hothersall Memorial Lecture, Blackpool, April 19, 1956. *Bull. Inst. Metal Finishing* (Trans. IMF), **6,** No. 2, 87-105 (1956).

Summary of complicated electrode phenomena associated with nickel, with a statement of classical thermodynamic limitations within which their mechanisms must operate. Principal features of Pourbaix potential-pH diagrams are explained, with special reference to the cathode and anode potential ranges as applied to practical nickel plating. Anode polarization and short-comings of theory with respect to behavior of nickel in the passive condition β are discussed. After a review of the electron configurations of nickel atoms and ions, mechanisms accounting for the polarization data are considered. Reaction steps proposed are such that the activation energy of the slow or controlling step can be reduced by H^+ ions, Cl^- ions and presence of certain impurities in nickel metal (reactive nickel). Potential-pH charts, polarization curves.—INCO. 13091

6.3.11, 6.3.12, 1.6

Investigations of Rhenium. C. T. SIMS, C. M. CRAIGHEAD, R. I. JAFFEE, D. N. GIDEON, W. W. KLEINSCHMIDT, W. E. NEXSEN, JR., G. B. GAINES, F. C. TODD, C. S. PEET, D. M. ROSENBAUM, R. J. RUNCK AND I. E. CAMPBELL. U. S. Wright Air Development Center Tech. Rept. 54-371, Supplement, September, 1956, 79 pp.

Arc-melted platinum-rhenium alloys containing up to 10 rhenium were studied for fabricability and properties. Results of resistivity, thermal emf, tensile strength and ductility, hardness and oxidation resistance determinations are given. Graphs show cold work-hardening characteristics for various types of rhenium compared with those of nickel and effect of rhenium, rhodium and nickel on the hardness of platinum. Tables, diagrams, 17 references.—INCO. 13086

6.3.14, 3.2.3

Determination of the Oxide Film Thickness on Tin. F. W. SALT AND J. G. N. THOMAS. *Nature,* **178,** 434-435 (1956) August 25.

The thickness of the natural oxide film on tin has been measured by electrometric reduction, the result being 15 Å after 1 hour of exposure of the fresh tin surface to air and 20 Å after 19 hours. The results are calculated on the assumption that the oxide film consists of stannous oxide.—BNF. 12924

6.3.14, 3.6.8

Studies on Corrosion of Tin. Rept. IX: Part II. Corrosion in Sulfuric Acid Solutions. G. YOKOYAMA. *J. Japan Inst. Metals,* **20,** No. 6, 344-347 (1956) June.

Corrosion of tin in sulfuric acid solutions below 30% is considered as a simple oxygen depolarizing type of corrosion. In acid above 30%, however, the influence of atmosphere change and the stirring effect on corrosion are not explained by the depolarizing mechanism. Various experiments were carried out and it was concluded that the cause of these abnormal corrosion phenomena may be ascribed to the depolarizing action of sulfate ions which are reduced on the surface of the corroding tin.—JSPS. 12999

6.3.15

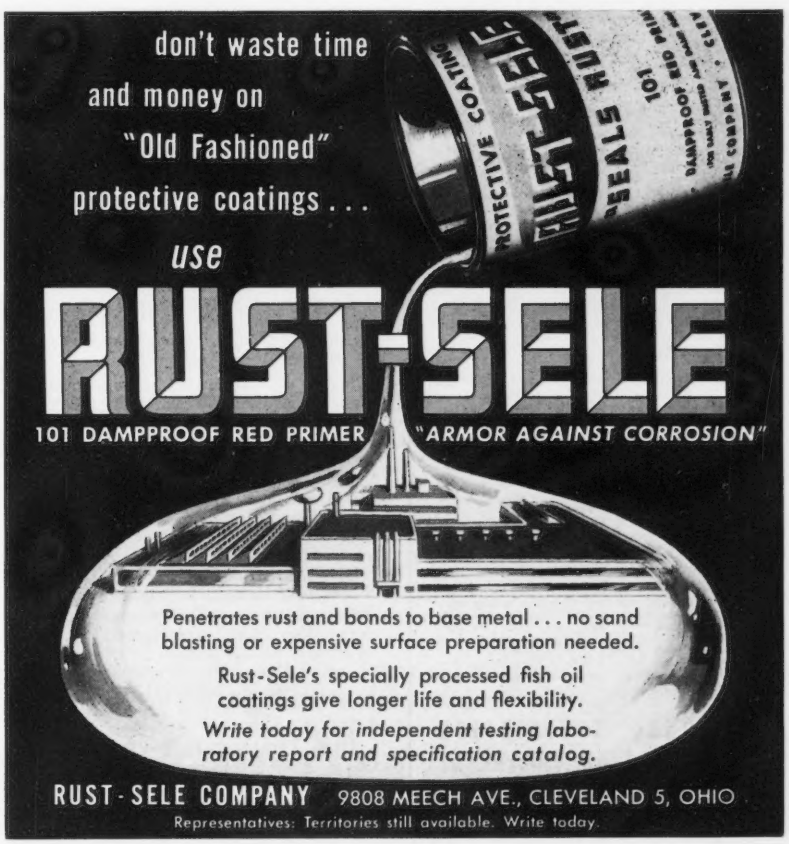
Studies of the Corrosion Resistance of Titanium Alloys. Part II. Corrosion Resistance of Titanium-Chromium, Titanium-Manganese, Titanium-Silicon and Titanium-Molybdenum Alloys. S. YOSHIDA, S. OKAMOTO AND T. ARAKI. *J. Mechanical Laboratory* (Japan), **10,** No. 2, 67-73 (1956) March.

The corrosion rates of titanium-chromium (5-15%), titanium-manganese (4-8%), titanium-silicon (0.5-1.5%) and titanium-molybdenum (5-30%) alloys were determined in various test solutions at ordinary and boiling temperatures. Alloys were arc-melted in argon atmosphere, hot and cold rolled to 1 mm thick plates and then cut into specimens for the corrosion tests. Results are summarized in figures and tables.—JSPS. 12998

6.3.15

The Electrochemical Properties of Titanium. Rept. I. Measurement of Spontaneous Potential and Corrosion of Titanium. S. MORIOKA AND A. UMEZONO. *J. Japan Inst. Metals,* **20,** No. 7, 403-407 (1956) July.

Spontaneous potential of titanium vibrates periodically to positive and negative directions in distilled or tap water, changing gradually to positive value with time, due to the positive and negative ions accumulating in anodic (pin holes of the oxide film) and cathodic (the oxide film) areas, respectively, and not being neutralized until they attain a certain amount in water. The spontaneous potential of titanium changes to a negative value with time in such solutions, under natural aeration, as hydrofluoric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acid >0.005%, hydrochloric acid >10%, sulfuric acid >5%, phosphoric acid >30%, ammonium fluoride >0.01%. On the contrary, in such solutions as nitric acid, chromium trioxide, potassium dichromite and potassium permanganate in acid solutions, Cu^{++}, Fe^{+++}, Ag^{+}, it varies largely to a positive value and titanium is passivated. The corrosion rate of titanium in naturally aerated hydrofluoric acid, sulfuric acid or hydrochloric acid solutions containing various oxidizing agents was measured.—JSPS. 12821

6.3.15

Transformation of Beta to Omega and Alpha in Titanium Alloys. (In German.) H. BOHM AND H. WESTPHAL. *Z. Metallkunde,* **47,** No. 8, 558-563 (1956) Aug.

The kinetics of the β to α transformation was studied by microscopy, hardness and dilatometry in three α/β Ti alloys (titanium-7 manganese, titanium-3 manganese-1 iron-1 chromium-1 molybdenum-1 vanadium and titanium-5 chromium-3 aluminum). Some alloys harden or become brittle under certain cooling or ageing conditions owing to the precipitation of an intermediate ω-phase.—BNF. 13014

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6.3.15

Corrosion of Titanium and Titanium Alloys. (In German.) O. RUDIGER, W. R. FISCHER AND W. KNORR. *Z. Metallkunde,* **47,** No. 8, 599-604 (1956) Aug.

Survey (11 references) was made of the corrosion behavior of unalloyed titanium. Surface layers which form in various corrosive agents were investigated by electron diffraction and mechanism of formation and separation of these surface layers was discussed. Potential-time curve of activated titanium in aqueous solutions and in air was determined and the influence of water on the passivation of titanium was established. By means of current density-potential measurements in 0.1 N fluorine-containing sulfuric acid, passivation potential of —0.6 V and activation potential of —0.4 V were established. Layers formed during anodic polarization were only partially identified. Secondary passivation was found from potential-time curves of titanium (prepassivated in water) in hydrochloric acid; this passivation layer, once formed, is confirmed up to 100 degrees, while primary passivation layer is dissolved at 40 degrees. Upper temperature boundary for completion of secondary passivation layer was obtained from steady potential at various temperatures and hydrochloric acid concentrations. Influence of alloying additions was studied. Definite improvement in corrosion resistance toward hydrochloric acid, sulfuric acid and phosphoric acid was found for molybdenum additions. An alloy with 30 molybdenum showed better corrosion resistance toward these acids than Hastelloy C. Graphs.—INCO. 12919

6.3.15, 3.2.2

Hydrogen Embrittlement of Beta-Stabilized Titanium Alloys. C. M. CRAIGHEAD, G. A. LENNING AND R. I. JAFFEE. *J. Metals* (Trans. AIME), Sec. 2, **8,** No. 8, 923-930 (1956) August.

Study included commercial or pure alloys of titanium with manganese and molybdenum. In α/β alloys tensile ductility falls with increasing hydrogen content, no hydride phase being visible in embrittled alloys. Embrittlement appeared to be of strain-ageing type.—BNF. 13010

6.3.15, 3.2.2

Effect of Hydrogen in Titanium Alloys. (In German.) H. W. SCHLEICHER AND U. ZWICKER. *Z. Metallkunde,* **47,** No. 8, 570-576 (1956) August.

Rates of absorption of hydrogen by titanium, titanium-6 aluminum, titanium-5 chromium-3 aluminum, titanium-4 aluminum-4 vanadium, titanium-7 manganese and a complex titanium-3 manganese alloy were determined. Creep tests to show the effect of hydrogen content. Preloading as well as hydrogen content and heat treatment affect the breaking load. Effect of hydrogen on the precipitation hardening properties of titanium-5 chromium-3 aluminum titanium-7 manganese and the complex titanium-3 manganese alloy was investigated to relate hydrogen embrittlement to the formation of the ω-phase. 16 references, nearly all to American work.—BNF. 12977

6.3.15, 3.5.9

High Temperature Tests on Commercially Pure Titanium. (In French.) G. SERTOUR AND M. EL GAMMAL. *Rev. Met.,* **53,** No. 8, 619-626 (1956) August.

Normal atmosphere oxidation tests show that titanium oxidation is important only above 600 C.—BTR. 12984

6.3.15, 3.5.9

The Oxidation of Titanium and Titanium Alloys. D. W. STOUGH, F. W. FINK AND R. S. PEOPLES. Battelle Memorial Inst. Titanium Metallurgical Lab., Columbus, Ohio (Contract AF18(600)-1375), January 30, 1956, 62 pp.

The oxidation of titanium and its alloys is reviewed. The scaling behavior in pure oxygen was found to vary according to three different rate laws in the approximate temperature ranges of 80 to 570 F, 570 to 1100 F and 1100 to 1800 F, respectively. The rate of oxidation increases with temperature; scaling becomes an important consideration in the fabrication and life of titanium materials above 1100 F. None of the commercial alloys were reported to have good oxidation resistance above 1100 F. However, experimental alloys with aluminum, silicon, tungsten, niobium, molybdenum and tantalum were found to scale less rapidly than the unalloyed metal between 1200 and 1800 F. Alloys with chromium, manganese, iron and vanadium generally were noted to scale faster than unalloyed titanium. (auth)—NSA. 12963

6.3.15, 3.5.9

The Oxidation of Titanium. (In German.) W. KINNA AND W. KNORR. *Z. Metallkunde,* **47,** No. 8, 594-598 (1956) August.

Investigation of structure of oxide films and mechanism of oxidation of titanium annealed in air or pure oxygen at 800, 1000 and 1200 C.—BNF. 12876

6.3.15, 3.6.5

The Standard Potential of Titanium. (In English.) SHUN-ICHI SATOH AND KOSHIN YAMANE. *J. Sci. Research Inst.* (Tokyo), **50,** 43-50 (1956) March.

The theoretical value of the standard potential of titanium is 1.30 v; an experimental value obtained for the standard potential of titanium in titanium sulfate solution is 0.355 v. The discrepancy between the calculated and observed values is attributed to the irreversible nature of the titanium electrode. The calculated value is accepted as the standard potential.—NSA. 12866

6.3.16, 3.5.9, 3.8.4

On the Oxidation of Tungsten at High Temperatures. (In Russian.) V. I. ARKHAROV AND IU. D. KOZMANOV. *Fizika Metallov i Metallovedenie,* **2,** No. 2, 361-369 (1956).

An experimental study of the phase composition of tungsten scales in the 500-1350 C range and the mechanism of oxidation at high temperatures.—BTR. 13112

6.3.19, 5.9.4

Preparing and Painting Zinc Surfaces. E. W. HORVICK. *Products Finishing,* **20,** No. 5, 36-38, 40, 42, 44, 46, 50 (1956) Feb.

Methods of preparing zinc surfaces for painting are outlined. They include alkaline cleaning, solvent cleaning, the use of chromate and phosphate solutions and sand or grit blasting. The permissible baking temperatures for finishes on rolled zinc and zinc die castings are indicated.—ZDA. 12778

6.3.16, 4.3.3

The Embrittlement of Tungsten by Ammonia. G. L. DAVIS. *Metallurgia,* **54,** No. 321, 18-20 (1956) July.

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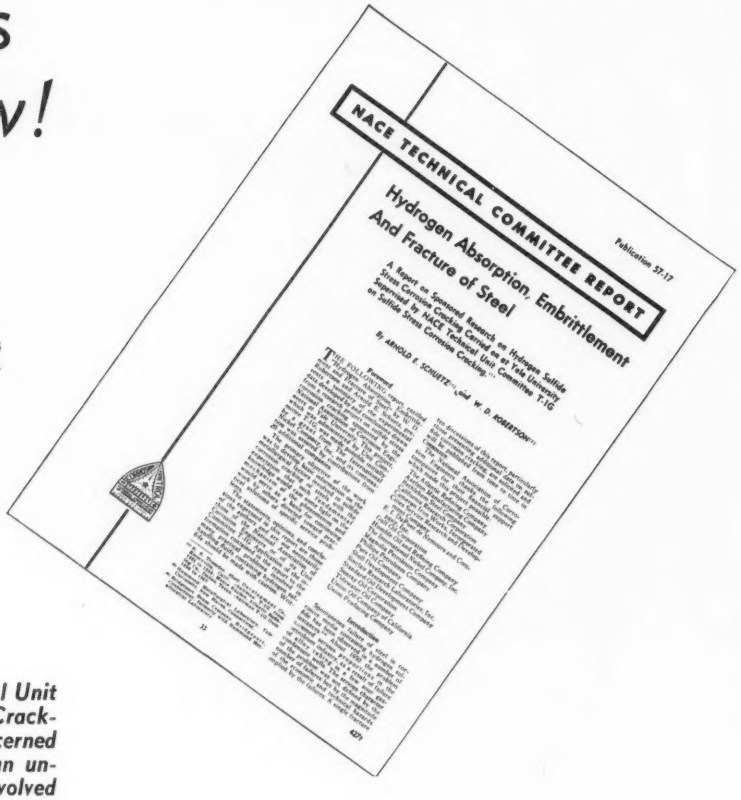

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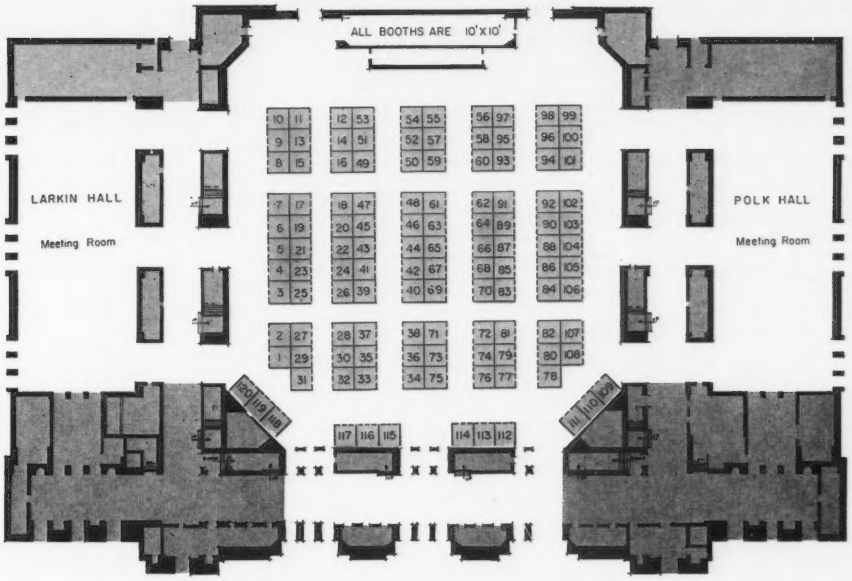

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anism governing embrittlement of tungsten when heated to a high temperature in an atmosphere containing small concentrations of ammonia (e.g., nitrogen/hydrogen/ammonia). Range of ammonia concentrations investigated was 0.01-20 vol. % at 600-2000 C for heating time of 0.1 to 20 minutes.—BNF. 13080

6.3.17, 3.2.3

Pilling and Bedworth's Law and the Phenomena of Attack of Uranium by Oxygen, Hydrogen, and Nitrogen. YVES ADDA. *Compt. rend.*, **242,** No. 1, 126-127 (1956).

Pilling and Bedworth's law [*J. Inst. Metals*, **29,** 529 (1923)] states that, if the ratio of the volume of oxide formed to that of metal >1, the oxide layer will be compact and act as a protection; if <1, the layer will be porous and the rate of attack will remain constant. Adda shows that this law does not hold with the oxidation, nitration, or hydridation of uranium, since, owing to the fissuration of the layers formed, there is no protective effect.—MA. 13084

6.3.17, 3.8.4

Some Observations on the Formation of Uranium Monoxide. J. WILLIAMS AND K. H. WESTMACOTT. *Rev. Met.*, **53,** No. 3, 189-204; disc., 204 (1956).

It is extremely difficult to obtain uranium monoxide other than as a film on the metal. A product containing 30% uranium monoxide has been obtained by heating uranium to 2000 C in a uranium dioxide crucible. Efforts to produce uranium monoxide in the massive form by the reaction of uranium dioxide with various reducing agents over a large range of temperatures and by partial oxidation of the metal were not entirely successful. It appears that a free surface is essential for the production and/or retention of appreciable amounts of uranium monoxide at room temperature. The structures observed suggest that the mononitride and monocarbide ac as catalysts. The growth of the uranium monoxide film can be explained by the diffusion of the uranium ion in both oxides. The uranium/uranium monoxide, uranium monoxide/uranium dioxide and uranium dioxide/atmosphere interfacial energies are probably important factors, but since the values of these free energies are not known, the exact part they play cannot be determined. UN can be distinguished from UC and uranium monoxide when they are all present as microconstituents in the metal, as it darkens very rapidly on heating in air, while the other two remain unaltered; UC stains easily when it is attacked by nitric acid-acetic acid-water mixture, while uranium monoxide dissolves but is not stained. 17 references.—MA. 13085

6.4 Non-Ferrous Metals and Alloys—Light

6.4.2

Corrosion of Aluminum-Magnesium Alloys. H. HARIMOTO. *J. Metal Finishing Soc. Japan*, **7,** No. 4, 134-140 (1956) July.

In previous papers (*Light Metals*, No. 3 (1951) May; No. 7 (1953) May; No. 8 (1953) August), the electrochemical properties of aluminum alloys were reported.

This paper deals with the electrode potential and the hydrogen evolution of 52S.

Samples were rolled alloy plates of 40%, 70% and 90% reduction, heated at 100 C and 200 C respectively. After heat-treatment, samples were dipped in 5%, 10%, 20% and 30% hydrochloric acid and the hydrogen-evolution was measured and then the character of oxide films produced on aluminum-magnesium alloys in 5% hydrochloric acid, 5% hydrochloric acid + 1% potassium chromate and 5% hydrochloric acid + 5% potassium chromate, was examined respectively by electron-diffraction method.—JSPS. 12777

6.4.2

Wear Life of Aluminium Gears. A. H. MASCHMEYER. *Product Eng.* **27,** No. 9, 160-166 (1956) Sept.

Tests of durability on gears in alloys 7075-T6, 2024-T42 with and without lubrication and anodized finish. Aluminum and aluminum, and aluminum on stainless steel. Theory for estimating probable life; stress modification factor (table of values for twelve combinations of materials being tested). Graphical presentation of results. Anodizing improved gear life with the chromic acid process usually giving best results.—BNF. 13067

6.4.2

Relation Between Electric Resistance Abnormality, Hardness and Stress Corrosion of Aluminum-Magnesium Alloys. H. YAMADA. *Light Metals* (Japan), No. 20, 55-57 (1956) August.

After solution treatment, the specimens of the aluminum-magnesium alloys containing practical amount of magnesium were tempered at 100, 150, 175, 200 and 250 C and electric resistance and hardness of each specimen were measured. An abnormal reduction of electric resistance and hardness were found in each specimen tempered at around 150 C.

The author investigated these abnormalities as well as the relation between the abnormalities and stress corrosion to which these alloys are apprehended to be vulnerable. The results obtained are as follows:

1) When the aluminum-magnesium alloys of supersaturated solid solution are tempered, those annealed at 150 C show abnormal reduction of electric resistance and hardness.

2) These abnormalities decrease with the addition of tin, lead, cadmium etc. to the aluminum-magnesium alloys.

3) These abnormalities do not seem to affect the stress corrosion.—JSPS. 13113

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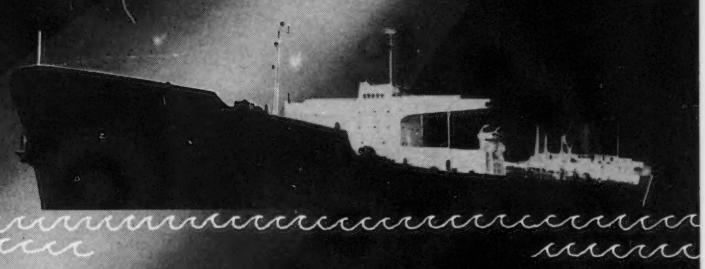

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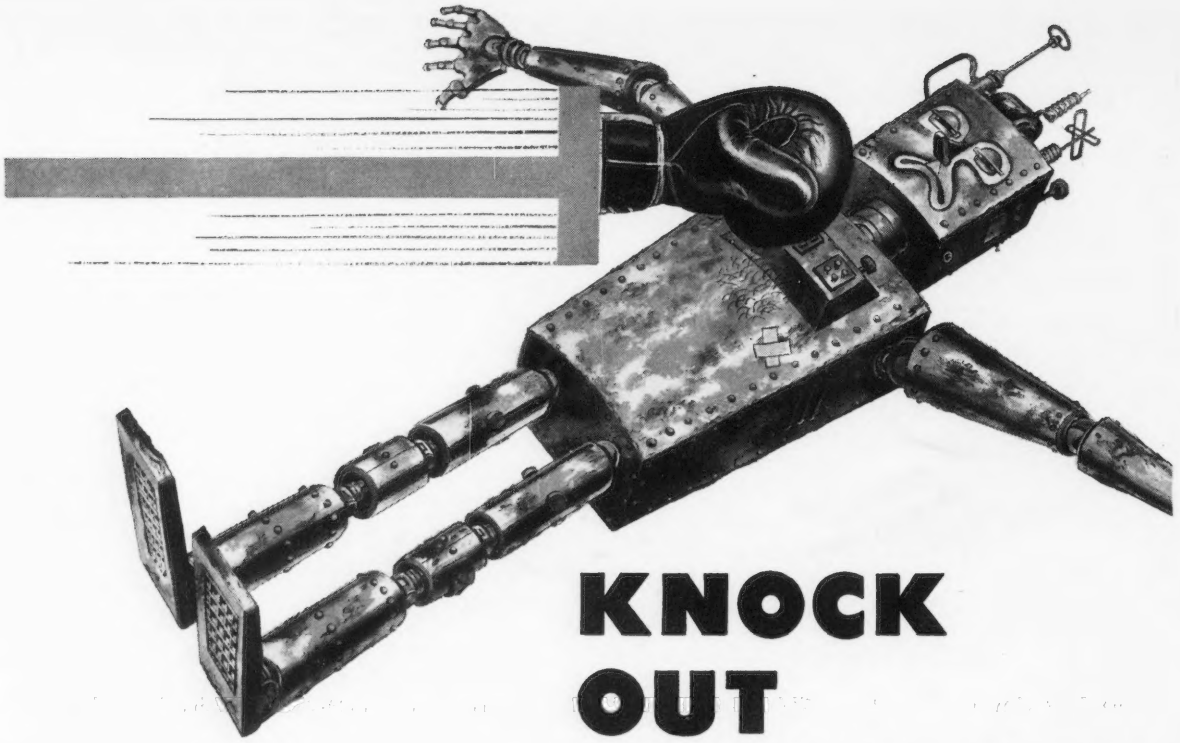

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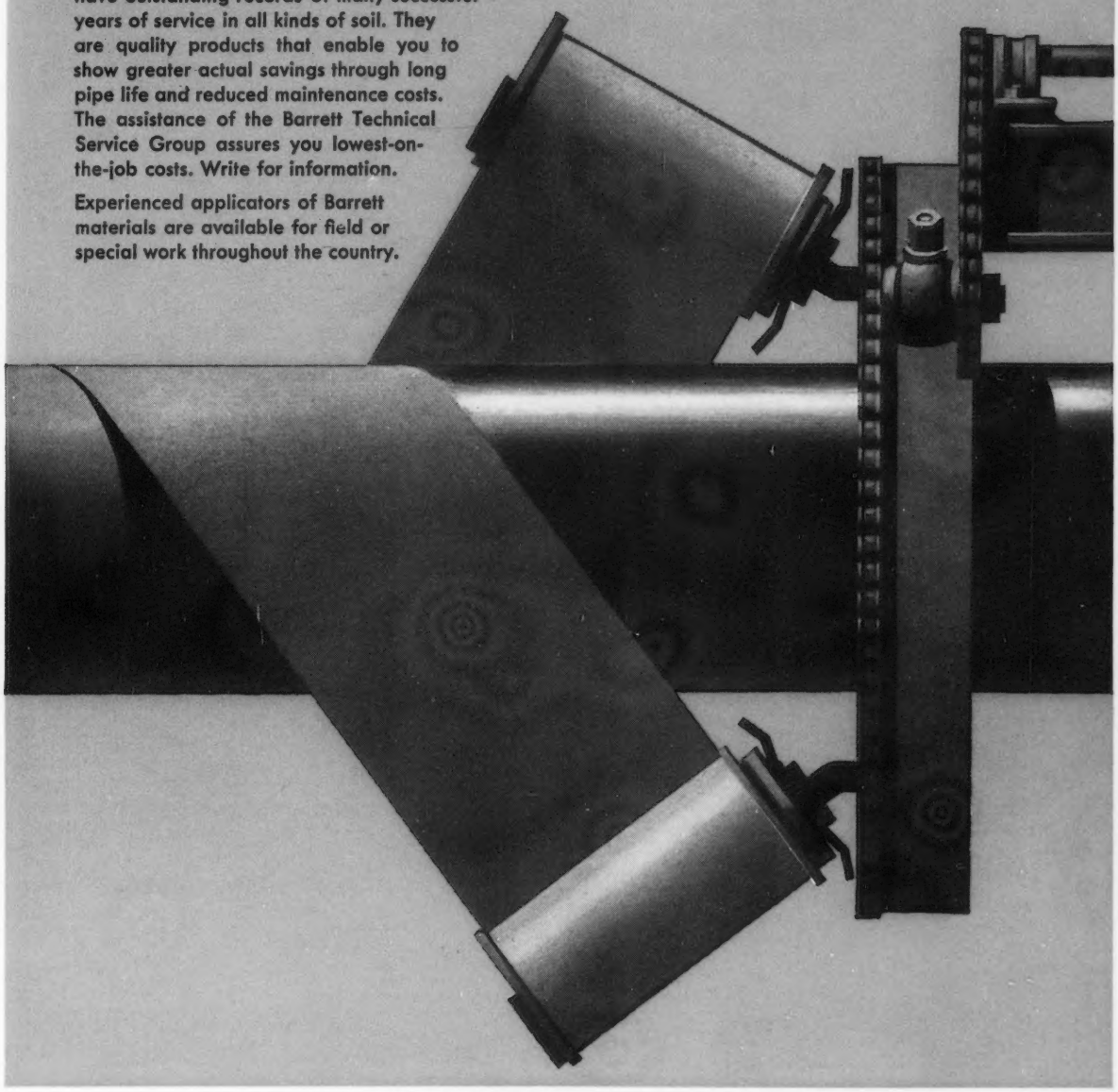

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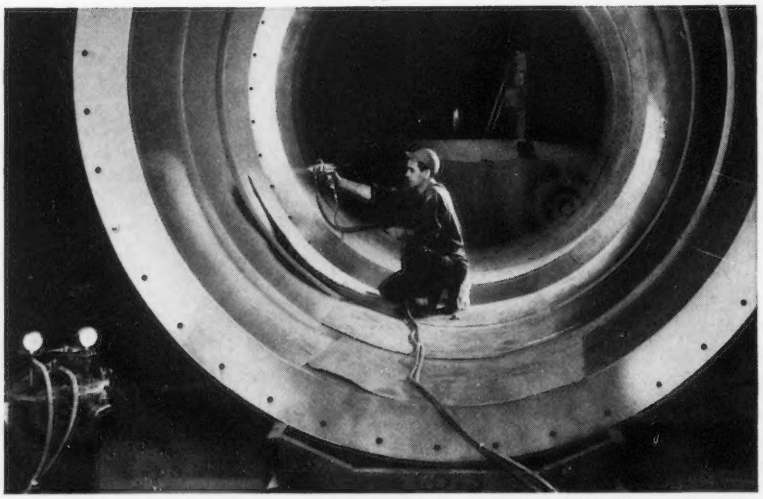

Large metal cones for storage tanks are sprayed with Unichrome "Super 5300" Plastisol Coating for protection against corrosion and abrasion. Photo Courtesy General Coating Co., Woodbridge, N.J.

Costs reduced, more durable linings produced with chemical resisting Unichrome Super 5300 Coating

Unichrome "Super 5300" Coating has established itself in the field of industrial linings. More and more companies are specifying this plastisol in place of sheet lining.

Sprayable Unichrome "Super 5300" Coating provides films up to 60 mils thick per coat. Multiple coats provide the thickness of plastic sheets, but are better because the plastisol conforms to irregular surfaces without air pockets or seams. "Super 5300" also saves money.

A HEAVY DUTY MATERIAL

Unichrome Plastisol Coatings demonstrate remarkable resistance not only to corrosion but also to abrasion. They withstand conditions that rapidly deteriorate unprotected metals or other materials. The coating has good flexibility to absorb mechanical abuse without harm. Should damage occur even years later, the coating can still be patched good as new. It sticks tight to primed metals – averaging a bond strength of 50 to 100 pounds per inch.

CORROSION-PROTECTION THAT ENDURES

Being a vinyl coating, a Unichrome Plastisol protects against acids, alkalies, and a host of other corrosives. The thick film blocks any permeation by solutions. So reliable is this protection, that over 1000 cleaning and metal finishing tanks in a single plant have not only been *lined* but also "lifetime" coated on their *exteriors* with Unichrome Plastisol.

Unichrome sprayable plastisols were the first to be available in colors. Specialists in various key locations are equipped to coat even large equipment. Tanks fabricated in sections can be coated and then bolted together after lining.

Informative bulletins available.

THE DURIRON COMPANY, INC./DAYTON, OHIO

ANODE DATA SHEET

Number 3

Fresh Water

Basic engineering information about DURIRON *impressed current anodes for cathodic protection.*

Protection of an Office Building Water Service Tank

THE PROBLEM: The cathodic protection of two 2600 gallon office building water service tanks. Aluminum anodes previously used in the installation had a comparatively short service life and required annual replacement. The continually increasing surface resistance of the aluminum anodes also required periodic rectifier adjustments to maintain an adequate protective current.

THE INSTALLATION: The tank is a single structure divided by a bulkhead to form two separate water storage units of identical size. The anodes were laid out to miss internal bracing, floats, and free-space for hatch opening. A single Duriron Type D (2" x 60") anode was first tested in parallel to fifteen 5 month old aluminum anodes (1-1/16" x 72") to determine its relative merits. Although the Duriron anode had slightly higher surface resistance when first installed, it showed no tendency for this surface resistance to increase and within 2 to 3 months was passing over twice as much current as the average current from the aluminum anodes.

After 311 days of the testing period the original anode proved so successful that all fifteen aluminum anodes (having reached the end of their useful life) were replaced with Duriron Type A (1" dia. x 60" long) anodes. One and one-half standard lengths were joined end to end making each anode assembly 7.5 feet long. Fifteen assemblies, together with the original test anode, were installed as shown in Fig. 2.

The test anode was inspected after more than two years of operation with the following results:

Total energy discharge—8700 ampere hours
Weight loss—3 ounces
Rate of loss—0.19 lbs. per ampere per year
Current density, over a 10 month period—0.14 to 0.21 amps per square foot

THE PERFORMANCE TO DATE: Five of the fifteen Duriron anodes installed on a permanent basis were inspected after 440 days of operation. The following table illustrates the results of this inspection.

Inspection Results After 440 Days Service

Anode Number	Total Energy Discharge (Ampere Hrs.)	Weight Loss (Ounces)	Consumption Rate Lbs./Amp./Yr.	Current Density (Amps per Square Foot)
1	3400	1	0.160	0.21 to 0.28
2	3181	½	0.086	0.20 to 0.27
3	3600	2	0.300	0.22 to 0.30
4	3000	¼	0.045	0.17 to 0.23
5	3400	1½	0.240	0.22 to 0.30

Incidental Data at Inspection Time:

A—Water resistivity in this tank was approximately 14,000 ohm-cm.

B—Average potential impressed on anodes was 25 volts, d-c.

A careful inspection was also made of the submerged joints of the five anodes. While a single neoprene washer appeared to adequately protect the submerged joint, polyethylene or polyvinylchloride tapes were also used over the joints to give further protection. All joints were found to be in excellent condition, although it was concluded that the taping of joints afforded little, if any, extra protection. There was no evidence of attack on the Monel inserts or the Everdur studs.

Corrosion attack on Duriron impressed current anodes is negligible at low current densities under the conditions of this installation. The Duriron anodes showed no appreciable increase in surface resistance during the entire period.

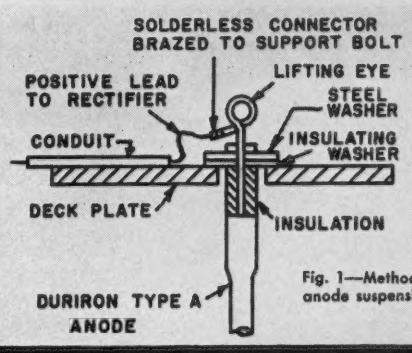

Fig. 1—Method of anode suspension

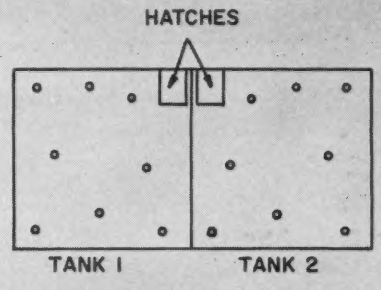

Fig. 2—Illustration of anode spacing

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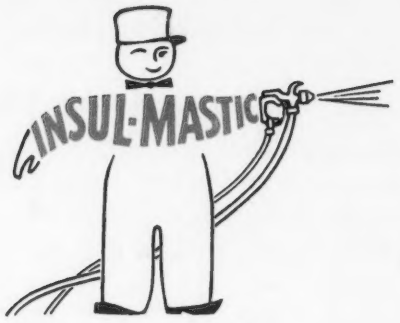

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